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FIRE RETARDANT BEHAVIOR OF KENAF FIBRE REINFORCED FLOREON COMPOSITE

By

LEE CHING HAO

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy AUGUST 2017

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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LEE CHING HAO

August 2017

Chairman: Professor Mohd Sapuan Salit, PhD, P.Eng
Faculty: Engineering

According to the report, more than 41% of fatalities in flight were to find to be caused by fire. In recent years, composites used in aircrafts are carbon fibre/ glass fibre reinforced epoxy, due to light weights and high strength properties. However, these composites are known as highly flammable. Serious fire incident will be created in a short time after a spark of fire. Furthermore, ingredients for fibre and epoxies are, toxic and resulting in the release of toxic gases during fire, and cutting off fresh air to survivors and hindering their escape. In the meantime, biopolymers have attracted considerable attention due to their environmentally friendly and sustainable nature, Kenaf Fibre (KF) is one of the most famous natural fibre used as a reinforcement in Polymer Matrix Composites (PMC). Kenaf is also known as *Hibiscus Cannabimus L.*, and is an herbaceous annual plant that is grown in a wide range of weather conditions, growing more than 3 meters within 3 months. However, the inherent drawbacks associated with Floreon (FLO) based composites include brittleness, lower strength and high moisture sensitivity, which in turn limit their application in the aircraft industry. In order to overcome such drawbacks, two modification techniques were employed in this study: (1) incorporated kenaf fibre into polypropylene polymer with magnesium hydroxide flame retardant and (2) reinforces kenaf fibre and magnesium hydroxide by different combination of volume. Consequently, KF reinforced FLO or polypropylene (PP) composites with magnesium hydroxide (MH) flame retardant specimens were successfully developed using extrusion followed by hot pressing. The increment of KF contents in PP composites had shown higher tensile modulus and decomposed mass loss at onset temperature, but lower values in tensile strength, elongation, flexural strength and onset temperature. In the meantime, 25 wt% KF contented PP composite shown a slightly higher flexural strength, while the higher volume of MH filler in composites caused lower strength, tensile modulus, elongation, but with higher onset temperature and the 2nd peak temperature in thermogravimetric analysis (TGA) testing. Furthermore, increasing the KF contents in PP matrix has found lower mass residue. However, increasing of KF contents in MH contented composite had increased the mass residue at the end of the testing. On the other hand, the increment of the melt flow properties (MVR and MFR) was found for the KF or MH insertion, due to the hydrolytic degradation of the polylactic acid (PLA) in FLO. The deterioration of the

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entanglement density at high temperature, shear thinning and wall slip velocity were the possible causes for the higher melt flow properties. In the meantime, increasing the KF loadings caused the higher melt flow properties while the FLO composites with higher MH contents created stronger bonding for higher macromolecular chain flow resistance, hence, recorded lower melt flow properties. However, the complicated melt flow behavior of the KF reinforced FLO/MH biocomposites was found in this study. The high probability of KF-KF and KF-MH collisions was expected and there were more collisions for higher fibre and filler loading, causing lower melt flow properties. Besides that, insufficient resin for fibre wetting, hydrolytic degradation on the biopolymer and poor interfacial bonding were attributed to low strength profile. Yet, further addition of KF increased the tensile strength and flexural. Nevertheless, inserting KF and MH filler have shown positive outcome on flexural modulus. Insertion of KF and MH showed the deterioration of impact strength, while the addition of KF increased the impact strength. Meanwhile, FLO is a hydrophobic biopolymer which showed only a little of total water absorption. In this regard, for the first 24 hours, the water absorption rates were high for all bio-composites. Hence, it is worth mentioning that the high contents of KF in bio-composites shown higher saturation period and higher total amount of water absorption while MH caused shorter saturation period but lower total amount of water absorption. However, interface bonding incompatibility has increased the water absorption of KF/FLO/MH composites. Moreover, some synergistic effect was located in char formation, T_g reduction and a lower tan δ peak shown in the three-phase system (KF/FLO/MH). The MH filler was found to be more significant in enhancing mass residual. The T_g were show deterioration for all samples compared to pure FLO biopolymer. The melting temperature has found no meaningful change for either insertion of KF or MH or both. The values of co-coefficient, C recorded decreasing as increasing the fibre loading. This showing the fibres transfer the loading effectively. As conclusion, although 10KF5MH specimen does not have the best performance in mechanical properties, a higher flame retardancy shall provide KF reinforced FLO composite with MH filler for more applications in advanced sector especially, in hazardous environment.

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Abstrak tesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai
memenuhi keperluan untuk Ijazah Doktor Falsafah

KELAKUAN RINTANGAN API BAGI KOMPOSIT FLOREON DIPERKUAT GENTIAN KENAF

Oleh

LEE CHING HAO

Ogos 2017

Pengerusi : Profesor Mohd Sapuan Salit, PhD, P.Eng
Fakulti : Kejuruteraan

Menurut laporan sebelum ini, didapati melebihi 41% kematian dalam penerbangan adalah disebabkan oleh api. Dalam kebelakangan ini, komposit yang digunakan dalam pesawat adalah serat karbon / gentian kaca dengan epoksi, kerana jisim komposit yang sangat ringan sertai sifat-sifat kekuatan yang tinggi. Walau bagaimanapun, komposit dikenali sebagai bahan mudah terbakar. Kejadian kebakaran yang serius akan terhasil dalam masa yang singkat dari percikan api. Di samping itu, bahan-bahan komposit yang dipakai di pesawat menghasilkan toksik dan mengakibatkan pelepasan gas toksik semasa kebakaran, dan mengurang udara segar yang diperlu oleh penumpang. Pada masa ini, biopolimer telah menarik perhatian oleh ahli sains kerana sifat mesra alam, Kenaf fibre merupakan salah satu fibre semula jadi yang paling terkenal yang digunakan sebagai tetulang dalam komposit polimer matriks. Kenaf juga dikenali sebagai Hibiscus Cannabimus L., dan merupakan tumbuhan yang tumbuh dalam pelbagai keadaan cuaca, ia tumbuh melebihi 3 meter dalam 3 bulan. Walau bagaimanapun, kelemahan telah didapati pada komposit Floreon termasuk kerapuhan, kekuatan yang lebih rendah dan lemah dalam kelembapan yang tinggi, oleh itu menghadkan aplikasi dalam industri pesawat. Dengan usaha untuk mengatasi kelemahan tersebut, dua teknik pengubahsuaian telah digunakan dalam kajian ini: (1) menambah kenaf fibre ke dalam polimer polipropilena dengan kalis api magnesium hidroksida dan (2) menggunakan kenaf fibre dan magnesium hidroksida dengan kombinasi yang berbeza kandungan. Spesimen untuk kenaf fibre /floreon atau polipropilena komposit disertai magnesium hidroksida telah berjaya dihasilkan dengan menggunakan penyemperitan diikuti oleh jentera panas menekan. Peningkatan kandungan KF dalam komposit polipropilena telah menunjukkan modulus tegangan dan kehilangan jisim pada suhu reput awal yang lebih tinggi, tetapi didapati nilai yang lebih rendah dalam kekuatan tegangan, pemanjangan, kekuatan lenturan dan suhu reput awal. Selain ini, 25% kandungan KF dalam polipropilena komposit menunjukkan kekuatan lenturan yang lebih tinggi, manakala kandungan MH yang lebih tinggi menyebabkan kekuatan yang lebih rendah, modulus tegangan, pemanjangan, tetapi dengan suhu permulaan yang lebih tinggi dan suhu puncak ke-2 dalam analisis Termogravimetri (TGA) ujian. Sebagai tambahan, meningkatkan kandungan KF di PP matriks telah menemui sisa kebakaran yang lebih rendah. Walau bagaimanapun, peningkatan kandungan KF di komposit berisi MH merunjukan meningkat sisa kebakaran pada akhir ujian. Sebaliknya, kenaikan sifat-sifat aliran leburan (MVR dan MFR) didapati apabila KF atau MH diisi, kerana kemerosotan hydrolytic asid polylactic (PLA) dalam FLO. Kemerosotan kepadatan tersangkut pada suhu yang tinggi, penipisan ricih dan slip dinding adalah sebab-sebab bagi sifat aliran leburan yang lebih tinggi. Pada masa itu, meningkatkan kandungan KF menyebabkan sifat aliran leburan yang lebih tinggi manakala komposit FLO dengan mengandungi MH yang tinggi menghasilkan ikatan kukuh bagi rintangan aliran rantaian makromolekul yang lebih tinggi, dan mencatatkan sifat aliran leburan yang lebih rendah. Walau bagaimanapun, aliran leburan yang rumit untuk KF/FLO / MH komposit didapati dalam kajian ini. Kebarangkalian perlanggaran yang tinggi untuk KF-KF dan KF-MH dijangkakan dan semakin tinggi perlanggaran untuk KF and MH dalam komposit, menyebabkan aliran leburan yang lebih rendah. Selain itu, resin yang tidak mencukupi untuk membalutkan fibre sempurna, degradasi hydrolytic pada biopolimer dan ikatan antara muka yang lemah telah menyebabkan profil kekuatan yang rendah. Namun, bagaimanapun, KF meningkatkan kekuatan tegangan dan lenturan. Selain itu, campuran KF dan MH telah menunjukkan hasil positif atas modulus lenturan. Campuran KF dan MH menunjukkan kemerosotan kekuatan impak, manakala penambahan KF meningkat kekuatan impak. Sementara itu, FLO adalah biopolimer hidrofobik yang menunjukkan penyerapan air yang rendah. Dalam tempoh masa 24 jam, kadar penyerapan air yang tinggi didapati untuk semua bio-komposit. Oleh itu, ia dicatatkan bahawa kandungan KF yang tinggi dalam bio-komposit menunjukkan tempoh tepu dan jumlah penyerapan air yang lebih tinggi manakala MH menyebabkan tempoh tepu lebih pendek dan jumlah penyerapan air yang lebih rendah. Walau bagaimanapun, ikatan yang ketidakserasian telah meningkat penyerapan air untuk komposit KF / FLO / MH. Selain itu, beberapa kesan sinergi terdapat dalam pembentukan char, pengurangan T_g dan puncak δ tan yang lebih rendah yang ditunjukkan dalam sistem tiga fasa (KF / FLO / MH). MH didapati lebih signifikan dalam meningkatkan sisa kebakaran. T_g menunjukkan kemerosotan untuk semua sampel berbanding tulen FLO biopolimer. Suhu lebur mendapati tiada perubahan yang ketara sama ada penggunaan KF atau MH atau kedua-duanya. Nilai-nilai bersama coefficient, C direkodkan berkurangan kerana peningkatan kandungan fibre. Ini menunjukkan fibre memindahkan beban dengan berkesan. Ahkirnya, walaupun 10KF5MH spesimen tidak mempunyai prestasi yang terbaik dalam sifat mekanik, tetapi rencat api yang tinggi menyediakan KF /FLO komposit dengan MH pengisi lebih banyak aplikasi dalam sektor maju terutamanya, dalam persekitaran yang berbahaya.

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APPROVAL SHEETS

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

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Universiti Putra Malaysia
Date:

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Declaration by graduate student

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

Signature:		Signature:	
Name of Chairman of Supervisory Committee:	PROF. IR. DR. MOHD SAPUAN SALIT	Name of Chairman of Supervisory Committee:	DR. MOHD ROSHDI BIN HASSAN

Signature:	
Name of Chairman of Supervisory Committee:	PROF. ALMA HODZIC

TABLE OF CONTENTS

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LIST OF TABLES

`

LIST OF FIGURES

`

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CHAPTER 1

INTRODUCTION

1.1 Introduction

In United State, in-flight smoke event happened once a day (Shaw, 2000). Fortunately, it has very low probability to become an uncontrolled in-flight fire. According to (Chaturvedi et al., 2001), more than 41% of fatalities are caused by fire in the air craft crash. From the statistic of 1987-2004, in-flight fires accident consumed top four leading fatalities beside of loss of control, controlled flight into terrain and specific component failure (Boeing, 2015). 423 fatalities accounted in 18 major in-flight fire accidents from 1990 to 2010 (Foundation, 2016).

Aircraft cabin fires generally grouped to ramp fires, in-flight fires, or post-crash fires. Ramp fires usually didn't threaten life because it occurs when aircraft is landed and parked. Therefore, escaping timing can be extending to more than an hour. In-flight fires normally detected when the beginning of fire and can be successfully extinguished. Yet it has some possibility to become uncontrolled fire. Post-crash fires are the most severe cases. Its scenario always started at landing (Galea and Markatos, 1987).

In year 2010, a tragedy post-crash fire happened, and 158 passengers were died. This involving in an Air India Express Boeing 737-800 when arriving in Dubai crashed as it overshot a “tabletop” runway in pre-monsoon rains and plunged into the jungle while trying to land according to TheGuardianNews. (McVeigh, 2010). The plane breaking into two immediately and burst into flames. Low visibility caused by two days of continuous raining and surrounded by hill made rescue works become harder.

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As a survivor describes the incident, he heard a loud bang and the plane caught in fire. After a while, the cabin filled with smoke hence visibility and oxygen levels are very low. The survivor survived by jumping out from the opening where the plane cracked. Only eight survivors have saved themselves in this incident.

On 11 July 1973, Varig Flight 860 landing at Orly airport when found out fire, the smoke filled the cabin quicky and propagated into flight deck (1973). Captain decided to call the landing emergency. The crew members were survived but did suffer in smoke inhalation.

From the experiences, in-flight fires normally started in inaccessible locations, and making extinguish process difficult to reach. All fire extinguishers work best when directly discharged on the fire source, but it is pointless to have it if we cannot reach that ignition spot. In Air Canada Flight 797, fire ignited behind the lavatory wall, people were known by growing smoke, but inaccessible to the fire (Board, 1986).

The most potential fire source is electrical. Two third of in-flight fires on Boeing were conducted by short-circuit (Aero, 2001). In a modern aircraft, there is over 150km of wire, any single point of wiring fault might cause uncontrolled fire (Potter et al., 2003). One example of wiring-fault-fire is Trans World Airlines Flight 800 B747 exploded on 3,900m height air. The main reason is because of sparking wire and led to explosion of centre fuel tank (Board, 2000). Increasing the thickness of wire insulation do helps to prevent mistake of wire. However, wire adds weight to aircraft. Therefore, manufacturers are keen to find lighter insulation materials.

Maintenance is very important to ensure a safety environment on aircraft. When the aircraft age is old, some of the black sticky debris is stick on wires which are flammable. When small sparks were dropped on these aging wiring, fast and uncontrollable fire occurs.

Besides, there are still a lot of fatalities caused by fire such as 1991 at Los Angeles International Airport (IRWIN, 1991). A fiery is collision on runway and burn into flames. Passengers had survived from the impact, but human crush happened due to low visibility caused by smoke and panic to rush to exit. From the fire scenarios testing, it has shown approximately 2minutes and 15seconds will grow to non-survivable conditions after fire ignition (Galea and Markatos, 1987).

In 1972, a United Air Lines B737 crashed at Chicago's Midway Airport. Accident report is showing high levels of cyanide in victim's blood stream and this is caused by toxic fumes inhalation (Transportation, 2000). Figure 1 shows the percentage of composite materials used in aircraft. Hence, the aim of this research is to improve the fire resistance properties of aircraft interior material. By doing this will allow passengers to have more time to escape from incident. Besides that, combustion of bio-composites produces lesser toxic gases compared to conventional composites.

From the above review, it is evident that were no previous work conducted on the mechanical and thermal properties of KF reinforced PP and Floreon composites with MH, which are used as flame retardant filler. Therefore, the aim of this present thesis is to find a high retardancy biocomposite materials.

1.2 Research Objectives

It cannot deny that fire is a major factor of accident fatalities. More survivors can be saved by improving the fire retardant of interior material. Therefore, improvement of fire resistance properties of the biocomposite material is done in this research.

The specific objectives of this project are as below:

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1. To fabricate the sample panel with different volume contents of kenaf fibre added into polypropylene and Floreon polymer.
2. To incorporate magnesium hydroxide flame retardant fillers into the kenaf fibre reinforced polymer composites.
3. To determine the melt flow properties of the bio-composites.
4. To determine mechanical properties (tensile, flexural, impact) of the bio-composites.
5. To determine the mass loss, modulus and other changes of the biocomposites across the temperature.

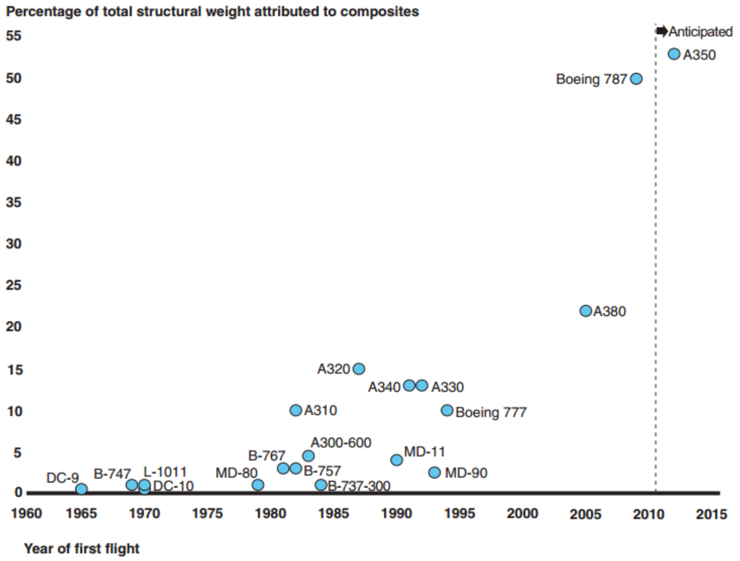

Figure 41. The percentage of composite materials used in aircraft.
(Source: Dillingham, 2011).

1.3 Scope of Study

The scope of work has covered several of fields. First of all, the selection of fibres and resins needed to be done. The properties of fibre-resin combinations are the most important in this research. Next study has included the fabrication of composites by different of method, curing time, and fibre-resin portion. Testing method had to concern to carry out the suitable experimental data for analysis. After that to determine the optimum combination of fibre-resin that having best fire retardant while remain sufficient strength for the composite.

In this study, Floreon has been selected as biomatrix. This is because Floreon is the latest advanced biopolymer invented by The University of Sheffield in year 2013. It is a biodegradable polymer which is constructed using standard polylactic acid (PLA). It was created for the greener, safer and better performance of the biopolymer. A lower manufacturing energy is required to produce FLO since it can be processed at about 160^{o}C, while most of the matrices require a temperature higher than 180 ^{o}C. Besides this, it ensures a lower chance of fibre thermal degradation, especially for a low thermal stability natural fibre. FLO is a recyclable and fully biodegradable polymer. Mechanical recycling, as in the case of polyethylene terephthalate (PET), is applicable to FLO. This method requires less energy to reproduce recycled plastic (52.6% less energy for recycling PET) as well as solving the landfill pollution problem.

1.4 Significant of Study

It is expected that the findings of this study may add to the effort to realize the potential of using kenaf fibre reinforced composites in development of new interior material for aircraft. Consequently, the new natural fibre composites can provide the flammability resistance to secure in aircraft fire incident. In addition, findings of this study may also help to enhance the knowledge in the new type of natural fibre composite materials. One of the application for kenaf fibre reinforced Floreon composite could be the interior panel in aircraft such as door panel. Door panel has generally experiences little force and it is the most important way in fire escape route. High flame retardancy of door panel can buys some valuable time for passenger to escape.

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1.5 Structure of the Thesis

This thesis is structured into 9 chapters. The first chapter is an introduction followed by Chapter 2 which presents comprehensive literature review on relevant areas associated with the topics in this research. The chapter 3 is the methodology chapter which showing the type of material used, preparation, characterization testing and flow chart.

The following Chapter 4 to Chapter 7 presents the findings of the research based on the alternative publication based thesis writing style. Chapter 4 encompassed the first article entitled "Mechanical and Thermal Properties of Kenaf Fibre Reinforced Polypropylene/ Magnesium Hydroxide Composites". The first article reported the use of non-biodegradable polymer as matrix for kenaf fibre and magnesium hydroxide additives, to study the properties of semi-biodegradable composite.

Chapter 5 presents the second article for the materials selection work entitled "Melt Volume Flow Rate and Melt Flow Rate of Kenaf Fibre Reinforced Floreon/Magnesium Hydroxide Biocomposites". This chapter discussed on the melt properties of fully biodegradable composite when kenaf fibre and magnesium hydroxide flame retardant was reinforced into Floreon.

Next is Chapter 6 which presents the third article entitled "Mechanical Properties of Kenaf Fibre Reinforced Floreon Biocomposites With Magnesium Hydroxide Filler". This chapter reported on the mechanical properties for fully biodegradable composite with different combination ratio of reinforcement kenaf fibre and flame-retardant magnesium hydroxide.

Chapter 7 presents the fourth article entitled "Thermal Analysis of Kenaf Fibre Reinforced Floreon Biocomposites with Magnesium Hydroxide Flame Retardant

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Filler”. This chapter deals with thermal properties of kenaf fibre reinforced Floreon composite with magnesium hydroxide flame retardant with different combination ratio. Finally, results and discussion are shown in Chapter 8 and in Chapter 9, overall conclusions and recommendations for future works are presented.

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CHAPTER 2

LITERATURE AND REVIEW

2.1 Introduction

Postcrash fire of an aircraft can be happen in minutes with appearance of flashover. A flashover spread about 1MJ of heat to surrounding and kills a lot of survivors. To delay or avoid the flashover incident, high fire-retardant materials should be considered. These materials able to pro-long the ignition time and thus passengers would have more valuable time to escape from cabin in fires. In this research, the main concerned is to find and create new composites that highly fire-retardant property. Therefore, before doing the laboratory or documentation works, it is important to do some research regarding to the topic. This included the knowledge of composite material, natural fibres, properties of kenaf fibres, and some information for fire retardant behavior as well as standard fire testing. These readings are originated from books, journals, and trustable online website.

2.2 Interior Panel of an Aircraft

Figure 2 shows standard interior panel of an aircraft by Lyon and Richard E. in their official aviation research regarding fire-resistant materials. The cabin components inside panel included ceiling, windows, lavatory, floors, partition, stowage bins, upper sidewall and lower sidewall. Besides, I had captured some real images from AirAsia aircraft AK1952 on Figure 3.

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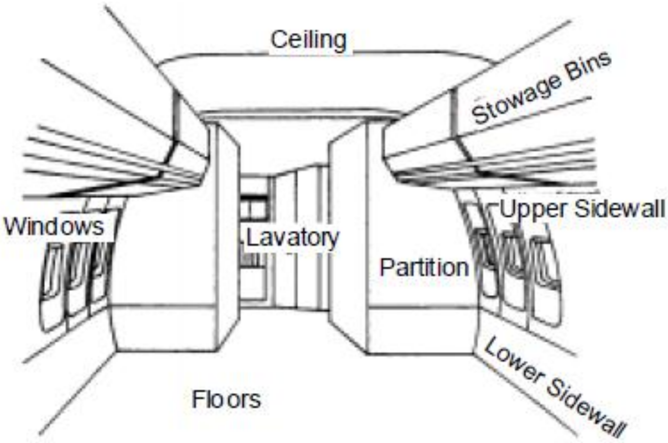

Figure 42. Cabin components.

(Source: Lyon, 1997)

Figure 43. Real aircraft interior at AirAsia AK1952.

Nowadays, most of cabin components had replaced by sandwiched structure that gives high strength and stiffness in low weight. Unfortunately, these advantages come along with highly flammable properties of composites. Weight of combustibles material in aircraft is recorded in Table ***122***1 and about total of 7000kg combustible cabin materials are in an average passenger aircraft. These polymeric materials release about

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35MJ/kg heat in fire (Lyon, 1997). Hence, 7000kg x 35,000kJ/kg = 2.5 x 10^8kJ of heat contained inside cabin area during fire. Worst case is this huge heat energy comes along with smoke and panic of human nature, eventually kills a lot of survivals.

Therefore, interior panel materials of an aircraft decided time frame for survival to escape from post crash fires, heat and smokes. Strong fire resistance materials postponed the ignition of fire, delay the burning period and at the same time reduce the smokes released. The materials that used to fabricate interior panel should fulfill following characteristics (Small et al., 2007),

a) Light weight
b) High fire resistance
c) High thermo-oxidative stability
d) Retention of material strength at elevated temperature
e) Does not release toxic smoke and extreme hat during combustion

2.3 Composite material in Aircraft Interior

Composite material is referred to a large number of strong, stiff fibres that surrounded by second different properties material known as matrix. By the way, there is another more accurate explanation for this research by Smith W. F. & Hashemi, (2006)

"*A composite material is a materials system composed at the best combination of two or more micro- or macro constituents with an interface separating them, which differ in form and chemical composition and are essentially insoluble in each other*".

The fibres can be found in form of natural, man-made, metallic, inorganic or organic. On the others hand, matrixes can be a metal or metal alloy, an inorganic cement, or a natural or even synthetic high polymer (Dietz, 1963). For various types of fibres and matrixes, a lot of possibilities combination could be produced depends on the situation needs. Table ***23***2 shows some examples of fibres, matrix and matching pairs.

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Table 122. Weight of combustible materials.

Cabin Material	Kilograms weight per aircraft, kg	Cabin Material	Kilograms weight per aircraft, kg
Acoustical insulation	**100-400**	Paint	**5**
Blankets	**20-250**	Passenger service units	**250-350**
Cargo liners	**50**	Partitions and sidewalls	**100-1000**
Carpeting	**100-400**	Pillows	**5-70**
Ceiling	**600**	Thermoplastic parts	**250**
Curtains	**0-100**	Seat belts	**5-160**
Ducting	**450**	Seat cushions	**175-900**
Elastomers	**250**	Seat upholstery	**80-430**
Emergency slides	**25-500**	Seat trim	**40-200**
Floor panels	**70-450**	Wall covering	**50**
Floor coverings	**10-100**	Windows	**200-350**
Life rafts	**160-530**	Window shades	**100**
Life vests	**50-250**	Wire insulation	**150-200**
		Total combustibles	**3300-8400**

Adopted from Lyon, 1997.

Composite can be found in some weapon to optimize its properties for example Japanese swords or sabers (Gay et al., 2003). The composite combination of sword is steel and soft irons. The steel is stratified like folding a sheet of steel over itself 15 times, 2^{15}= 32768 layers, with orientate the defects and impurities in long direction. Soft iron is being added after this. Good impact resistance is the feature of this composite combination.

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Table 23. Example of fibres, matrix and matching pairs.

Fibre	Matrix	Matching pairs (good adhesion)
Asbestos	Phenol-formaldehyde	Asbestos/phenolic
Glass	Melamine-formaldehyde	Asbestos/melamine
Carbon	Polyester	Glass/polyester
Aramid	Epoxy	Glass/epoxy
Boron	Silicone	Glass/silicone
Silica	Polyimide	Aramid/epoxy
	Polybenzimidazole (PBI)	Carbon/epoxy
	Furane	FC/silica
	Friedel-Crafts (FC)	Boron/polyimide
		Boron/PBI

Adopted from Dietz, 1963.

2.3.1 Advantages of composites

In reality, it is hard to find a specific material which can meet all requirements for all situations. Therefore, Engineer combines two or more materials into one that has superior properties than individual material to achieve some specific requirements (Smith and Hashemi, 2006).

Lighter weight compared to other material is a feature of the composite. Commercial aircraft keep searching for composite material because the lighter weight of the craft leads to fuel saving, increase in payload and improves. Approximately 15% of total automobile weight being strikes out by replacing the composite part (Mohanty et al., 2002).

Besides that, large parts of product like aircraft wings can be moulded directly and separately. This will reduce the use of tooling and quality time. Composites normally

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do not yield because of their elastic limits corresponding to the rupture limit (Gay et al., 2003).

Composites can withstand in humidity for a long period, (Gay et al., 2003) said that epoxy resin can absorb water by diffusion up to 6% of its mass while composite of reinforcement/resin can absorb up to 2%. Next, composites have a great corrosion resistance. Hence most of the product that placed in extremely situation is made by composites. Composites also have excellent fire resistance when compared with light alloys with identical thicknesses. But the smokes emitted from certain matrixes might be toxic. However, Composites materials of interior aircraft that going to research in this thesis are bio-composites which only a little toxic gas when fire incident happened.

2.4 Natural Fibres

As environmental issues have come to the fore, the use of natural fibre reinforced biopolymer composites has become a priority in all sectors. Over the last few decades, biocomposites have undergone a remarkable evolution. Research is increasing every year in the area of biocomposites, starting from 32 papers in the year 1997 and dramatically increasing to 716 papers in the year 2015 in the journal paper website ScienceDirect. Figure 44 shows the statistics of biocomposite related topics in ScienceDirect from the year 1997 to the year 2015. Positive increases were indicated for each year, meaning that biocomposites research has become increasingly famous due to the environmental issue. Many researchers are keen to seek a solution to substitute for the conventional materials that are harmful to our world.

The good flexibility, high stiffness, and low cost of the biocomposites make them the priority selection for users. Besides this, the limited supply of petroleum has made

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biocomposites even more popular. Therefore, intensive research has been conducted to develop biocomposites that are compatible with conventional products (Mohanty et al., 2000; Bledzki et al., 2002; Mohanty et al., 2005; John and Thomas, 2008; Pickering, 2008; Satyanarayana et al., 2009; Thomas and Pothan, 2009; Hassan et al., 2010; Summerscales et al., 2010; Venkateshwaran and Elayaperumal, 2010; Shinoj et al., 2011). This review aims to discuss the natural fibres and biopolymers that have been most studied. However, not all types of natural fibres and biopolymers can be covered in this review, and only a few have been selected for discussion.

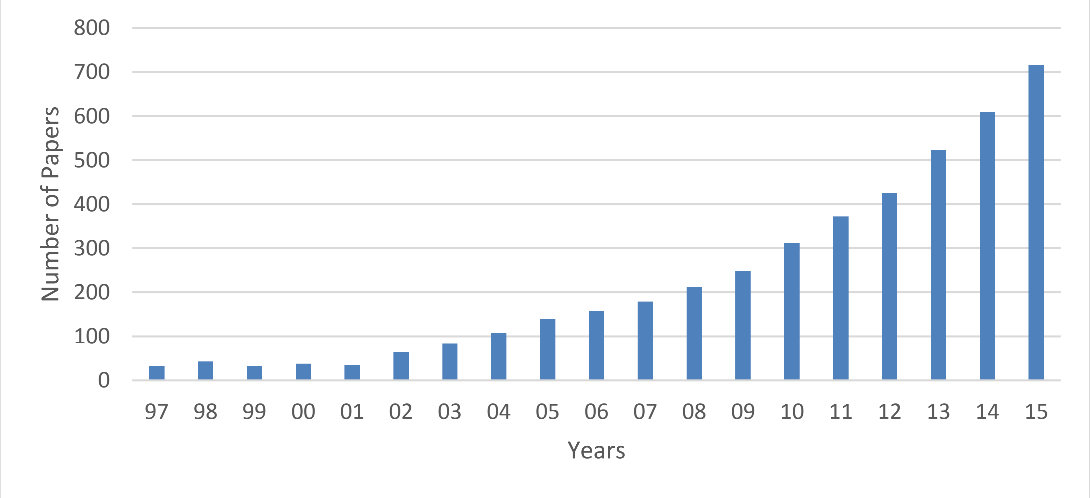

Figure 44. Statistics of biocomposite related topics in ScienceDirect in the years 1997 to 2015.

The automotive, construction, aviation and other sectors are keen to substitute biocomposites for heavy, weak or expensive materials (Pothan et al., 2003). Natural fibre is a renewable resource and it will substitute all traditional materials in the future. Natural fibre can be categorised into plant, animal and mineral types, as illustrated in Figure 45. As there is a higher demand for superior material nowadays, surface modification has been applied to enhance the properties of natural fibre so that the modified natural fibre reinforced biopolymer composite is able to perform well in

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advanced sectors. In recent times, cellulose fibre has undergone the most intense research in the previous five years period. Figure 46 shows the natural fibre that has been used in biocomposite related topics in ScienceDirect during the years 2011 until 2015. Cellulose fibre constitutes about a quarter of the total natural fibre used in biocomposite research. Apparently, cellulose fibre has outstanding strength properties as it is constructed of pure cellulose.

The advantages of natural fibre can be seen in most of the research journals that relate to it; many researchers have mentioned these advantages in their works (Anuar and Zuraida, 2011;Elfehri Borchani et al., 2015). Table ***324***3 states the properties of some natural and synthetic fibres.

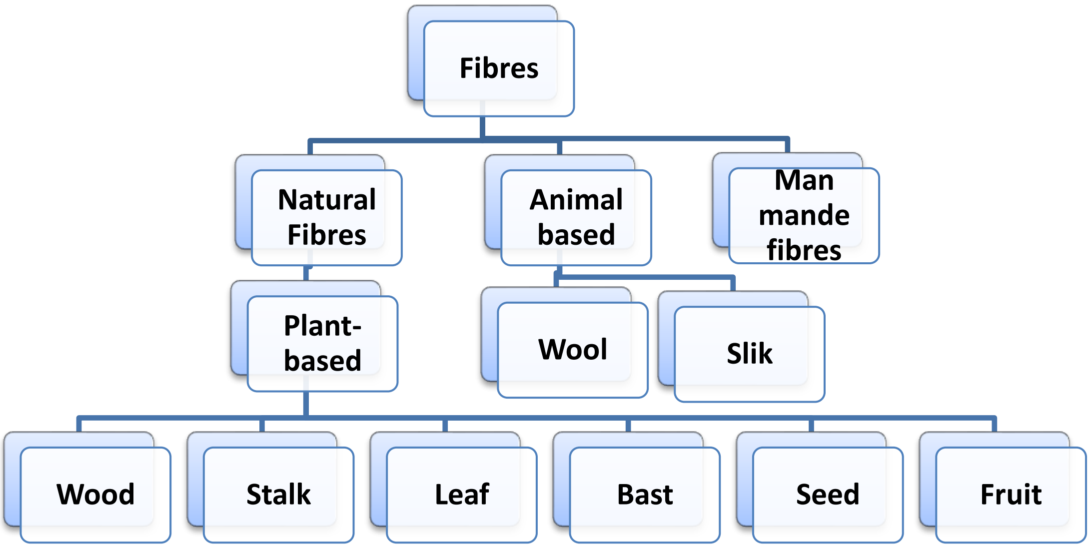

Figure 45. Grouping of the natural fibres.

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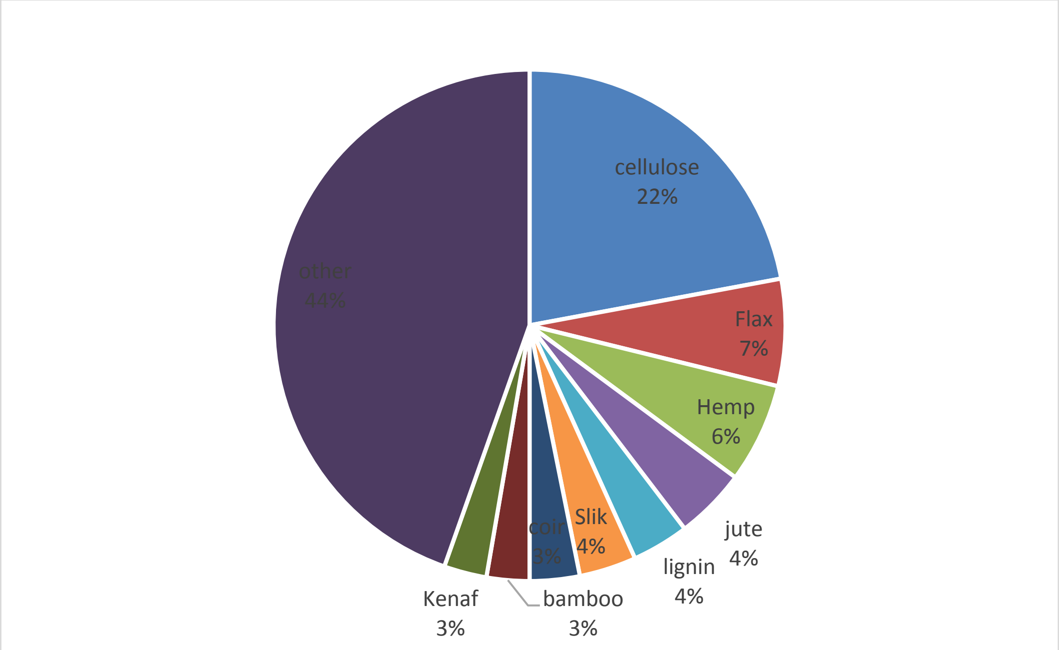

Figure 46. The natural fibres used in biocomposite related topics in ScienceDirect in the years 2011 to 2015.

Table 324. The properties of some natural and synthetic fibres.

Fibre	Density (g/cm^3)	Elongation (%)	Tensile Strength (MPa)	Elastic Modulus (GPa)
Cotton	1.5	7	400	12.6
Jute	1.3	1.8	773	26.5
Flax	1.5	3.2	1500	27.6
Hemp	1.47	4	690	70
Kenaf	1.45	1.6	930	53
Ramie	-	3.8	938	128
Sisal	1.5	2.5	635	22
Coir	1.2	30	593	6
Softwood kraft pulp	1.5	4.4	1000	40
E-glass	2.5	0.5	3500	70
S-glass	2.5	2.8	4570	86
Aramid	1.4	3.7	3150	67
Carbon	1.4	1.8	4000	240

Adopted from Ku et al., 2011.

Pineapple leaf fibre and banana leaf fibre are naturally occurring waste products (Ho et al., 2012). These products are going to be thrown away before humans have noticed their usage. Therefore these natural fibres are available at very low cost compared to synthetic fibres (Jawaid and Abdul Khalil, 2011).

With respect to the environmental issue, biodegradable natural fibre can reduce the problem of solid waste yield and handling matter (Jawaid and Abdul Khalil, 2011). Other than the landfill issue, energy consumption is another aspect of environmental concern. A lower energy is required to produce the same amount of the natural fibre compared to the synthetic fibre. It is only takes 15MJ of energy for 1kg of the kenaf fibre, while glass fibre consumes 54MJ (Akil et al., 2011). Natural fibre has lower densities of 1.2 – 1.6g/cm^3 than 2.4g/cm^3 of glass fibre (Huda et al., 2006). A lower density of natural fibre has more volume or quantity of fibres for the same weight. This scenario means that natural fibre is fabricated with much lower energy, but with a much higher quantity of fibres. Beside this, natural fibre is non-abrasive to equipment; this helps to prolong the life period of machine tools and reduces the maintenance cost (Akil et al., 2011). The environmentally friendly production process of the natural fibre offers better working conditions to the workers and reduces the risk of respiratory problems compared to intrapleutral fibrous glass which causes chest pain, troubled breathing, sore throats, and coughs (Newball and Brahim, 1976;Jawaid and Abdul Khalil, 2011). Natural fibre shows a good damage tolerance and better elongation when a load is applied to it (Jawaid and Abdul Khalil, 2011). Spider silk is an animal based natural fibre that is gathered during the web making process (Ho et al., 2012). Spider silk allows more than 200% elongation of its original length, and it needs about triple the amount of kevlar fibre breaking energy to break the silk (Bonino, 2003).

However, a low interfacial bonding between the natural fibre and the polymer reduces the properties and performance of the composites. The void in the composite turns itself into a stress concentration point and a crack propagation starting point(Amato et al., 2011). Several methods have been introduced to modify the natural fibre surface to improve the fibre-matrix interfacial such as a coupling agent, pre-impregnation and graft copolymerisation (Herrera-Franco and Valadez-González, 2004).

Hydrophilic in nature, the natural fibre is incompatible with the hydrophobic polymers (Alvarez et al., 2004;Baiardo et al., 2004;Akil et al., 2011). The water absorption behaviour of the natural fibre leads to fibre swelling at the fibre matrix interphase (Mehta et al.,2004). This may cause a decrease in the mechanical properties of the composite (Ku, et al., 2011). High water absorption is no doubt the main disaster for the natural fibre reinforcement composite.

Expelling the dye compounds in the wastewater into clean water will dissolve the oxygen in the water and will prohibit sunrays from passing through the water (Sajab et al., 2011). A variety of methods can be used in removing dye compounds for example, adsorption (Rafatullah et al., 2010), membrane filtration (Amini et al., 2011) or electron-catalytic degradation (Ma et al., 2009). The conventional method shows an effective result, yet another form of solid waste has been produced. Therefore, researchers have found cheaper and potential absorbents in natural fibre. The high water absorption of natural fibre is a useful feature in dye compound removal in wastewater (Hassan, 2015).

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2.4.1 Chemical Component of Natural Fibres

“Lignocellulosic fibre” is a scientific name that refers to natural fibre, because all plant fibres are constructed of just a few constituents (cellulose, hemicellulose, and lignin). Most of the plant fibres contain 50-70% cellulose as shown in Table 4.

The structure of a plant’s cell wall is known as a microfibril (Figure 47). The natural fibre is constructed of millions of macrofibrils, while a macrofibril is framed by micofibril that consists of cellulose, hemicellulose and lignin (Akil et al., 2011;Ho et al., 2012). Each of the macrofibrils consists of an outer layer of primary cell wall and three inner secondary cell walls. Lumen is the region of open empty spaces located at the centre of the macrofibril, and it reduces the bulk density of the natural fibre.

Fibre	Cellulose	Hemicellulose	Lignin	Extract.	Ash Content	Water soluble
Cotton	82.7	5.7	-	6.3	-	1.0
Jute	64.4	12.0	11.8	0.7	-	1.1
Flax	64.1	16.7	2.0	1.5-3.3	-	3.9
Ramie	68.6	13.1	0.6	1.9-2.2	-	5.5
Sisal	65.8	120	9.9	0.8-0.11	-	1.2
Oil palm EFB	65.0	-	19.0	-	2.0	-
Oil palm Frond	56.0	27.5	20.48	4.4	2.4	-
Abaca	56-63	20-25	7-9	3.0	-	1.4
Hemp	74.4	17.9	3.7	0.9-1.7	-	-
Kenaf	53.4	33.9	21.2	-	4.0	-
Coir	32-43	0.15-0.25	40-45	-	-	-
Banana	60-65	19	5-10	4.6	-	-
Sun Hemp	41-48	8.3-13	22.7	-	-	-
Bamboo	73.83	12.49	10.15	3.16	-	-
hardwood	31-64	25-40	14-34	0.1-7.7	<1	-
Softwood	30-60	20-30	21-37	0.2-8.5	<1	-

Besides this, the lumen is widely deployed as an acoustic and thermal insulator in nature. On the other hand, the microfibril in the secondary cell wall is composed of crystalline cellulose or crystalline hemicellulose and amorphous lignin, arranged alternately with a width of 5-30nm (Baillie, 2005). Each layer of microfibril is implicated in the designated angle to hold the fibre from every direction.

Cellulose is the main constituent of all the plant fibres (Chawla, 2005). It is composed of C, H, and O_2 elements with a formula of $C_6H_{10}O_5$. Cellulose also influences the major characteristics of plant fibre. It is highly hydrophilic in nature due to the hydroxyl groups (-OH) that are found in the cellulose chain (Baillie, 2005).

Hemicellulose is the most abundant material after cellulose. Hemicellulose contains highly branched chains and is built from several sugars such as glucose, glucuronic acid, mannose, arabinose and xylose (Summerscales et al., 2010). They form hydrogen bonding with cellulose and covalent bonding with lignin. Lignin is a highly complex amorphous structure. The lignin acts as a cementing material and fills the spaces between the cellulose and the hemicellulose (Mohanty et al., 2002). The functioning of the natural fibre components is shown in Table ***526***5.

Table 425. Chemical content of natural fibre.

Fibre	Cellulose	Hemicellulose	Lignin	Extract.	Ash Content	Water soluble
Cotton	82.7	5.7	-	6.3	-	1.0
Jute	64.4	12.0	11.8	0.7	-	1.1
Flax	64.1	16.7	2.0	1.5-3.3	-	3.9
Ramie	68.6	13.1	0.6	1.9-2.2	-	5.5
Sisal	65.8	120	9.9	0.8-0.11	-	1.2
Oil palm EFB	65.0	-	19.0	-	2.0	-
Oil palm Frond	56.0	27.5	20.48	4.4	2.4	-
Abaca	56-63	20-25	7-9	3.0	-	1.4

Hemp	74.4	17.9	3.7	0.9-1.7	-	-
Kenaf	53.4	33.9	21.2	-	4.0	-
Coir	32-43	0.15-0.25	40-45	-	-	-
Banana	60-65	19	5-10	4.6	-	-
Sun Hemp	41-48	8.3-13	22.7	-	-	-
Bamboo	73.83	12.49	10.15	3.16	-	-
hardwood	31-64	25-40	14-34	0.1-7.7	<1	-
Softwood	30-60	20-30	21-37	0.2-8.5	<1	-

Adopted from Jawaid and Abdul Khalil, 2011

2.5 Kenaf fibre (*Hibiscus Cannabimus L)*

Kenaf fibre is one of the more famous natural fibres used as a reinforcement in Polymer Matrix Composites (PMC). Kenaf is known as *Hibiscus Cannabimus L.,* and is an herbaceous annual plant that is grown in a wide range of weather conditions, growing more than 3 meters within 3 months (Nishino et al., 2003). The highest growth rate may be up to 10 cm/day. However, the difference in growth parameters influences the properties of the kenaf fibre, e.g. length of the growth season, plant population, cultivar, planting date, photosensitivity and plant maturity. The stem of the kenaf plant is straight and is not branched along the stem. It is built up of bark and a core. Therefore, it is easy to separate the stem by either chemicals or enzymatic retting. The bark contributes 30-40% of the dry weight for the stem, while the wood-like core makes up the remaining weight. The long bast fibre type is used to make composite boards, textiles and pulp and in the paper industry.

Table 526. Chemical content and function of natural plant fibres.

Chemical content	Polymeric state	Molecular derivatives	Function
Cellulose	Crystalline, highly oriented large molecule	Glucose	Fibre

Hemicellulose	Amorphous, smaller molecule	Polysaccharides, galactose, mannose, xylose	Matrix
Lignin	Amorphous, large 3-D molecule	Phenyl propane, aromatic	Matrix
Extractives	Some polymeric and non-polymeric	Fat, fatty acid, phenols, terpentes, waxes	Extraneous

Adopted from Sapuan S. M. et al., 2009.

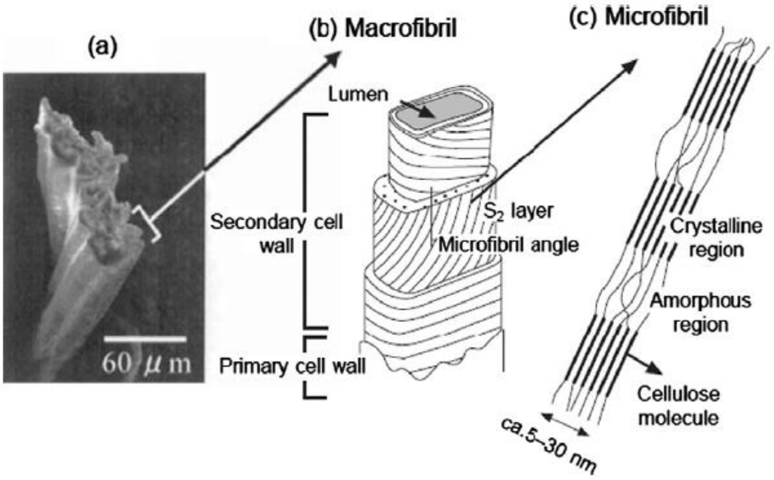

Figure 47. Evolution of natural fibre (a) scanning electron micrograph of kenaf bark fibre (b) macrofibril (c) microfibril of natural plant.

(Source from Baillie, 2005).

Rouison (Rouison et al., 2004) has revealed the two main attractions of the kenaf fibre. Firstly, the kenaf plant absorbs the nitrogen and phosphorus in the soil. These minerals help to increase the cumulative weed weight, crop height, stem diameter and fibre yield. Kuchida (Kuchinda et al., 2001) suggested that the nitrogen application at 90kgN/ha has a significant effect for kenaf plant growing. The other attraction is the high photosynthesis ability of kenaf (Nishino, Hirao et al., 2003). The tripled photosynthesis rate of kenaf (23.4mg CO_2/dm^2/h) compared to conventional tress (of 8.7mg CO_2/dm^2/h) under 1000ų mol/cm^2/s helped to reduce carbon dioxide while producing oxygen (Lam et al., 2003).

The lighter and porous kenaf core fibre is rich in hemicellulose and lignin content (Alireza A., 2003). It is reported to have a better bonding ability than bast fibre since

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the lignin acts as a cementing agent in the fibre (Paridah M. T., 2009). Kamal (Izran Kamal, 2009) has made a new particleboard using kenaf core fibre with polypropylene (PP). The performance of the particleboard is satisfactory, except for its high flammability, which is caused by the nature of the kenaf fibre and the petrochemical polymer product. Therefore some modifications have been done to the board in order to solve this issue. A few several types of fire retardant filler (DAP, MAP and BP® (Boron)) have been added into the sample and tend to achieve better results. Untreated particleboard only took 50 seconds to ignite, while the BP® was able to extend its ignition period to 2 minutes. Besides this, it has only 8.52% of the burnt area with 0.69% of the weight loss. The boron is capable of providing protection to postpone the heat transfer (Horrocks and Price, 2001). Furthermore, other fire retardant filler candidates were chosen to conduct the same investigation, hoping to get better results (Aisyah et al., 2013).

The bast kenaf fibre has better strength properties than the core fibre, hence it is more suitable for high strength applications. One study has used kenaf bast fibre to reinforce a concrete composite in order to compare its properties with plain concrete (Elsaid et al., 2011). The results indicated that the mechanical properties of the concrete composite were comparable to the plain concrete specimen. Furthermore, the concrete composite has shown an evenly distributed cracking and higher toughness. Therefore, the concrete composite is claimed to be a potential material for construction applications.

The automotive sector has used natural fibre reinforcement composites in its designs for decades to achieve lower fuel consumption, lower cost, and better environmental friendliness. However, the poor mechanical properties of the renewable materials has hindered the idea of using natural fibres. Davoodi (Davoodi et al., 2010) has focused on combining kenaf and glass fibre to improve the properties for car bumper beams. The promising mechanical properties of this hybrid composite material have shown the potential of the natural fibre in the automotive sector. On the other hand, 5 design conceptions of the kenaf fibre polymer composite have been used in the automotive parking brake lever introduced by Mansor (Mansor et al., 2014). One of the concept designs was selected for further development. Several selection processes and computerised analysis were performed to replace the existing heavier steel-based parking brake lever, while maintaining the strength and performances.

2.6 Biopolymer

The application of natural polymers began in ancient times (Thomas et al., 2013). However, the ease of availability of petrochemical plastics at a lower cost and with better properties means that they have replaced natural polymers. Nevertheless, many biodegradable polymers are found in the market nowadays due to the high awareness about environmental pollution and the shortage of fuel supply. Biodegradable refers to the capability of the polymer to decompose into methane, water, carbon dioxide, inorganic compounds and biomass. The biodegrading mechanism is caused by biological activity, mainly by the enzymatic action of micro-organisms. Biodegradable materials are widely used in packaging and medical applications.

Most of the synthetic polymers today are produced using petroleum and they are non-biodegradable. The stable, long life duration of the synthetic plastic waste has become a serious pollution issue for groundwater and solid waste. To recycle the plastic waste, extra work and energy is needed to clean up and recycle the materials. On the other hand, incinerating the plastic waste causes global warming by increasing the carbon dioxide content in the air.

With the increasing awareness of ecological preservation, biodegradable polymers are reducing the dependence on the limited availability of petroleum and the burden of the carbon dioxide footprint. The use of biopolymers in various industries brings an economical advantage due to the lower cost of the biopolymer raw material. In addition, the higher usage of renewable resources helps to sustain the economy by increasing the income of the agricultural sector (biopolymer and natural fibre). Furthermore, the used biopolymer can be further utilised as a bio-fuel to generate a power supply without the emission of toxic gases during the combustion process that damage the environment.

The low strength properties are the main obstacle of biodegradable resins. It is not advisable to use the bio-resin directly for structural components, especially for high performance requirements. The biodegradation process is indeed an ideal for plastic waste, yet positing a serious threat to plastic-in-use, a drawback for biopolymers to be used in high duration environments. A hot water transfer pipe that was constructed using synthetic polyvinyl chloride (PVC) is highly stable under high temperatures, but thermal degradation happened on the biopolymer. This has shown that the biopolymer has a very limited working temperature window, which prohibits it from many applications. Fortunately, biopolymers can enhance the properties by the addition of plasticisers (more flexible), flame retardant filler (better flame resistance), and fibre (stronger) and many other ways.

Different classifications of biodegradable polymer have been introduced and can be classified according to their synthesis method - either obtained or synthesized from renewable resources (Figure 48). Figure 49 shows the classifications of polymer studied in biocomposite related topics in ScienceDirect in the years 2011 until 2015. The polysaccharides biopolymer undergoes the highest amount of research. Starch is

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one of the most famous polysaccharides polymers. Polylactic acid (PLA), polyhydroxyalkanoates (PHA) and polyvinyl alcohol (PVA) are the main representatives for the category of bio-synthesis from biomass, microbial hydrolysis from biomass and chemical-synthesis from fossil product, respectively. Conventional polymers still occupy a significant position in research, mainly being used to compare performance with biopolymer composites, showing that biodegradable polymers are capable of application in advanced sectors.

Matrixes playing a very important role on composite as it distribute the load evenly to the reinforcement fibre and it has lower density and modulus of elasticity than fibre (Dietz, 1963).Without matrix, fibres act nothing in composites. Appendix 1 listed behavior of thermo-set and thermoplastic in term of odor, color of flame, burning speed, and smoke characteristic.

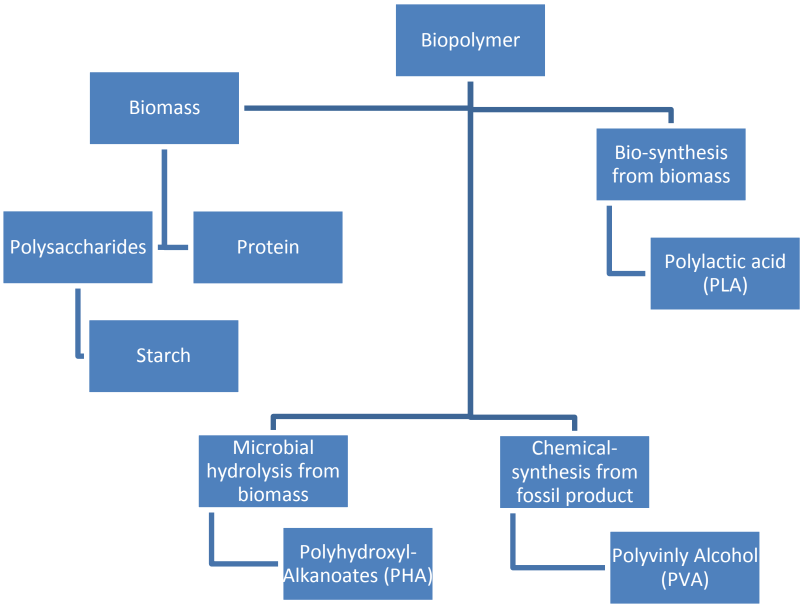

Figure 48. Classification of the main biodegradable polymers.

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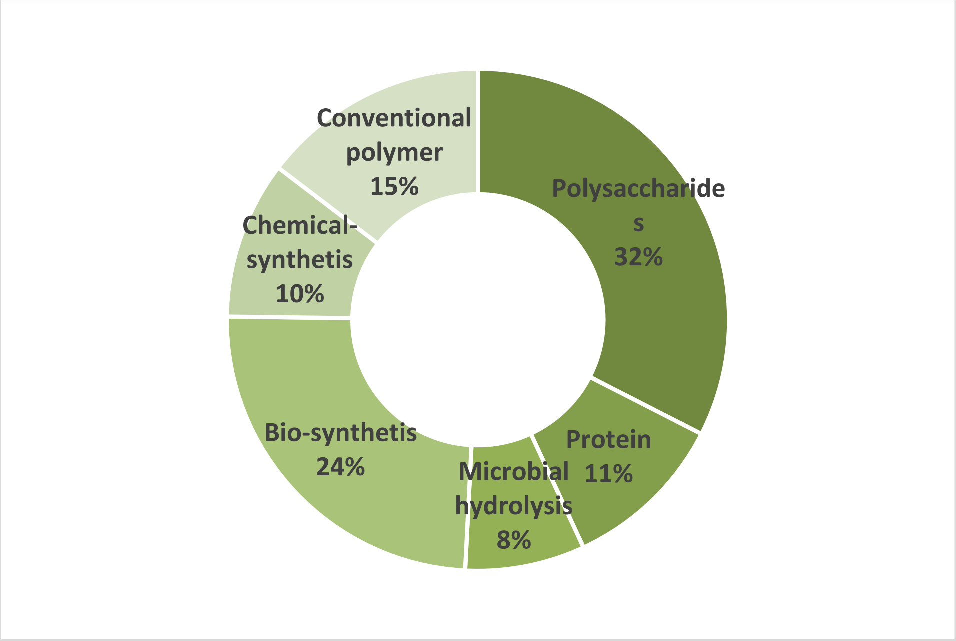

Figure 49. The classification of polymers studied in biocomposite related topics in ScienceDirect in the years 1997 to 2015.

Polypropylene is a thermoplastic polymer used widely in variety of field such as stationery, plastic part, and packaging (Sain et al., 2000). In 2008, 49.6 million tones of polypropylene had consumed by world market. These statistics contribute a lot of landfill waste since polypropylene is non-degradable. Therefore some business men convert the waste polypropylene into higher economic value as well as reduce land pollution by adding natural fibre become bio-composites (Suharty et al., 2012). Installing Kenaf natural fibre into polypropylene can actually completely improve its properties and biodegradability (Premalal et al., 2002;Kim et al., 2005;Suharty and Firdaus, 2006).

Polypropylene made by propylene monomer which is hydrocarbon gas (-CH2=CH(CH$_3$)-) from petroleum refining (Gibson and Mouritz, 2006). Chemical structural of propylene and polypropylene noted in Figure ***50***10. As you can see there is a double bond bonding first and second carbon. To form long chain polymer, this double bond being open up and connected with neighbouring monomer.

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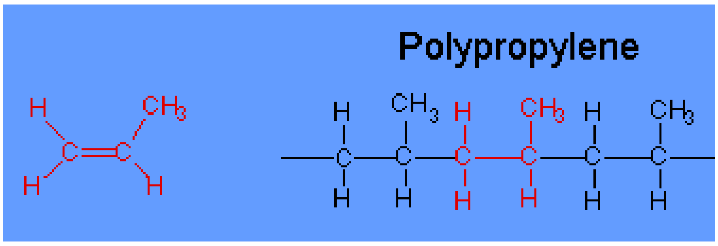
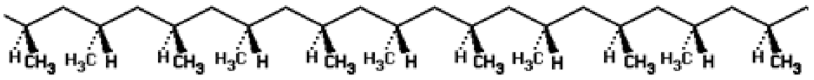


Figure 50. Chemical structural of propylene and polypropylene.
(Source Ophardt, 2003).

Besides that, orientation of methyl group (-CH_3) does influence the characteristic of polymer. When all the side chain methyl groups arranging in regular orientation along the polymer chain, it is known as isotatic polypropylene. This stereoregular structure allowed maximum intermolecular forces and hence it is considered as strongest polypropylene. Another similar strength of syndiotactic polypropylene formed by regularly alternation of hydrogen and methyl groups in front and back of the polymer chain. Third and last type of amorphous type called atactic polypropylene. It has random arrangement of side chain methyl groups in the long polymer chain. Therefore it naturally softer and flexible than other two forms. Three forms of chemical structure illustrated in Figure 51 (Brown, 2012).

Isotactic Polypropylene

Syndiotactic Polypropylene



Atactic Polypropylene

Figure 51. Three forms of chemical structure.
(Source Brown, 2012)

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2.6. 1 Polylactic acid (PLA)

Polylactic acid (PLA) is not a new biopolymer in research. It is a member of the aliphatic polyester family, made from α-hydroxy acid which has extraordinary properties comparable to fuel-based polymers like polypropylene (Henton D. E.., 2005) and polystyrene (Fang and Hanna, 1999). It has extensively matured in the last decade and has gone into mass production for commercialise uses (Glasbrenner, 2005). The monomer, lactic acid (LA), is produced by the bacterial fermentation process of renewable resources such as corn, which effectively reduces the dependence on fuel-based polymers (Garlotta, 2001). The PLA is a white powder with about 175^{o}C of T_m and 55^{o}C of T_g. Three isomeric forms, L-, D- and meso-LA can be found in the fermentation result. L- and D-LA are isomers and they play a significant role in the properties of the final product (Figure 52). The behaviour of PLA is dependent on the polymer structure chain and the ratio of L- and D-LA in the polymer (Witzke D. R., 1997;Hartmann, 1998). PLA synthesis is performed by polycondensation and ring-opening polymerisation (ROP) methods. Polycondensation produces a low molecular weight PLA polymer, therefore some modifications and additives are needed in the process for a better molecular weight, but this does increase the cost at the same time. The ROP method has produced a high molecular weight PLA, but highly purified, raw, lactide material is needed.

The properties of PLA are greatly influenced by the L-lactic content inside the polymer. 100% of L-lactic PLA polymer has around a 45-70% crystalline index, and lower crystalline indexes and lower T_g for lower L-lactic content. On the other hand, the increment of the D- lactic contents causes the PLA polymer to shift to amorphous material with lower T_m. Amorphous classification is tagged on the PLA polymer when the L-lactic content is less than 87.5%.

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The PLA polymer can biodegrade completely into water and carbon dioxide (CO_2) in aerobic conditions (rich in oxygen). Dry and pure PLA polymer can last for more than 10 years, whereas in practice the moisture in the surroundings contacts the PLA surface and under hydrolysis. Therefore the half-life of the semicrystalline PLA polymer in a normal situation lasts for several months while the amorphous PLA polymer only lasts for several weeks (Kharas G. D., 1994).

The PLA polymer usesa less energy in production, and is produced from renewable sources. It requires less than 25-55% of the energy to produce the PLA by the ROP method (Xiao L., 2012). Furthermore, its greater thermal processabillity has made it a good alternative green material (Rhim et al., 2006). The company of Cargill Dow has claimed that using biomass sources and wind power to produce the PLA polymer means that extremely low energy is consumed (Vink et al., 2003).

However, several reasons have caused the PLA polymer to be retarded from becoming fully substituted for the fuel-based polymer. The high brittleness of PLA (<10% elongation at break) and its high production cost have made it poor in performance (Rasal and Hirt, 2009).

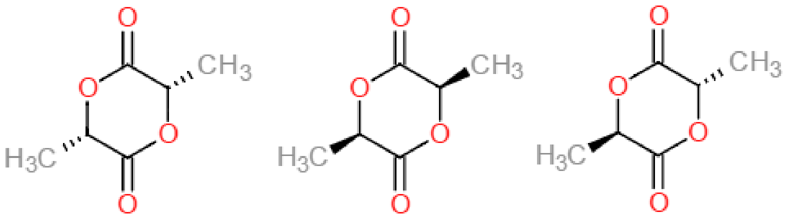

Figure 52. The chemical structure of a) L-lactide, b) D-lactide and c) meso-lactide.

2.6.1.1 Synthesis of PLA

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The synthesis method is a crucial step in producing PLA with superior properties. Direct polycondensation and ring-opening polymerisation (ROP) are the most popular synthesis methods for producing the PLA polymer. However, industrial production is preferred to a multi-step process by combining both methods, as mentioned in a previous paper (Garlotta, 2001).

The LA monomer is produced by the fermentation of abundant carbon sources with suitable micro-organisms under anaerobic conditions (Huang, 2005). Research has showed ineffective LA production under oxygen-rich conditions (Hammes and Hertel, 2006). Hence most of the fermentation process is carried out without oxygen. The micro-organism produces ATP energy (adenosine triphosphate) and a reducing agent (NADH) by converting the glucose (carbon sources) into pyruvate. After that, the reducing agent further reduces the pyruvate into the more reduced LA.

However, not every micro-organism can produce the pure lactic acid for high molecular weight PLA, therefore strain selection has to be very carefully considered (Hammes and Hertel, 2006). Besides this, the fermentation of LA gradually converts the medium into an acid-based medium until it is unable to continue due to the strong acid environment. Calcium carbonate is a straightforward way to neutralise the acid medium, but it fails to produce pure LA as a large amount of the side-product gypsum ($CaSO_4.2H_2O$) is found in the neutralisation reaction. Therefore, a purification process is highly necessary.

The direct polycondensation method has become famous due to it being a relatively simple and cheap method. It extracts the water molecules by condensation and produces one equivalent of water for every single reaction (eq1). There are difficulties in water removal due to the increment of viscosity during polymerisation, and this produces a low molecular weight PLA polymer. Applying a vacuum environment is found to enhance the water removal but leads to the formation of ring structures of different sizes (Kéki et al., 2001).

$$HO\text{-}(CH(CH_3)COO)_n\text{-}H + HO\text{-}(CH(CH_3)COO)_m\text{-}H > HO\text{-}(CH(CH_3)COO)_{n+m}\text{-}H + H_2O$$

Equation 1(Kéki et al., 2001)

The condensation process produces a low molecular weight polymer with hydroxyl- and carboxyl-end chains. The chain extension method is a solution which involves adding a chain coupling agent to link the hydroxyl- or carboxyl-low molecular weight PLA polymers (Xiao L., 2012). The final product can be purely hydroxyl- or carboxyl-terminated PLA using either a small amount of multifunctional hydroxyl compounds (glycerol, 1,4-butanediol) or multifunctional carboxylic acids (maleic acid, succinic acid). Besides this, it can be done by melting with a small amount of a chain coupling agent so that the additional cost can be ignored, but the impurities of the final product are always a challenge to fabricators.

Azeotropic condensation polymerisation, a condensation method, has resulted in a PLA end-product with a higher molecular weight. Distillation of the LA is performed for 2-3 hours at 130^{o}C under reduced pressure firstly to remove the condensed water. A high content of residual catalyst causes unfavoured degradation. Therefore, a further filtration is needed with the addition of strong acids to reduce the residual catalyst.

Considering high molecular weight PLA polymer production, the ROP synthesis method is introduced by the LA dimer, with lactide as a cyclic ester, and the synthesis process begins by opening the ester bond on the ring. Metal cation mechanisms help to accelerate the polymerisation in low temperatures but it induces some degradation (oxidation, hydrolysis) (Witzke D. R., 1997;Hartmann, 1998). Hence it should be used in a very small amount. Lewis acids and alkali metal alkoxides are good cationic catalysts (Kricheldorf et al., 1995;Degée et al., 1999). On the other hand, anionic lactide polymerisation is performed by the nucleophilic reaction of the anion with carbonyl and acyl-oxygen cleavage. A relatively high temperature (120 ^{o}C) is needed to initiate the reaction.

The ring-opening method was introduced in 1932, but high molecular weight PLA polymer was not achievable until 1954; DuPont demonstrated the lactide purification process (Garlotta, 2001). Under reduced pressure, the depolymerisation of the low molecular-weight PLA yields a mixture of L-lactide, D-lactide and meso-lactide, and the mixture configuration depends on the types of feedstock, temperature and catalyst. Back-biting and end-biting reactions are found in the process (Yoo et al., 2006). The back-biting reaction refers to the intra-molecular reaction between the carboxylic group and the ester backbone for the formation of a cyclic compound, while end-biting refers to the ring closure reaction of a linear chain. The main difference between these reactions is that end-biting produces a cyclic compound and water, while a cyclic and a linear compound is produced by back-biting.

2.7 Fire Retardant of Composites

Safety precaution is a requirement of every designing product. Fire is the most general topic even in safety of composites. Sustainable combustion/ burning process require all the three elements: oxygen, fuel, and heat. To stop a fire, termination of any element will do and normally is decrease the concentration of oxygen. Somehow existences of natural fibre in composites ameliorated its overall properties, but it limited to the ability against fire. This is explainable as per natural fibres are plant or animal base product. Cellulose, hemicelluloses and lignin are more likely to fire compared to polymer plastic.

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The interior panel of aircraft is usually made of fire retardant composite. Polypropylene can burns very rapidly with a relatively smoke-free flame and without leaving a char residue (Zhang and Horrocks, 2003). This is a major benefit and reason to make polypropylene as interior panel materials of an aircraft since smoke killed a lot of survivor in history. High flammability of polypropylene allows it has a rapid decomposite rate at high self-ignition temperature (570^{o}C).

In year 2000, Shemwell do some research on soot release by polypropylene compare to other four plastics. Polypropylene produces very low smoke emission when excess oxygen is provided (Shemwell and Levendis, 2000). On the others hand, cool flame combustion at 350^{o}C conducted by other researcher says it could leads to formation of toxic compounds (CO gas) which can cause death in sudden.

Modification allows altering properties of material, same thing goes to flame retardancy of polypropylene. Fire retardant composite is the composite added with inert fillers or thermally active fillers to the polymer matrix. Fire retardants are categorized into additives compounds and reactive compounds. An acceptable flame retardant for polypropylene should have following features,

1. It should be thermally stable up to the normal polypropylene processing temperature (<206^{o}C).
2. It should be compatible with polypropylene and have no leaching and migratory properties.
3. The additive should retain its flame retardant properties when present in the fibre.
4. It should also reduce the toxicity of gas and smoke during burning to an acceptable level.

5. It should be present at a relatively low level to minimize its effect on fibre properties as well as cost.

Nevertheless, hundreds type of filler and thousands type of combination exist in market. Each of combination gives different result. Table ***627***6 extracted the list of flame inhibiting compounds conducted previously.

Suharty N. S. (2006) had concluded that kenaf fibre reinforced polypropylene composites have 0.56 second longer time to ignition rather than pure polypropylene which using 1.6 seconds. Besides that, composites installed with fire retardant compounds are future postpone the ignition time to 3.22 seconds. This shows a tremendous success of using fire retardant fillers. On the other hands, highly flammable polymer can be modified to slower burning rate by adding kenaf fibre. Burning rate of pure polypropylene and its composites is 2.15 and 1.09 mm/min respectively. Furthermore, a flame inert barrier can be created by adding fire retardant compounds and it was proven to reduce until 0.55 mm/min.

Table 627. List of flame inhibiting compounds conducted.

List of Flame inhibiting Compounds	References
Ammonium salts of sulfate Phosphate	(Tesoro, 1978)
$CaCO_3$ nano-particles	(Parta et al., 2005)
Diammonium phosphate Ammonium sulfate	(Liodakis and Antonopoulos, 2006)
Magnesium carbonate SnO_2 coated with $CaCO_3$	(Xu et al., 2006)
Diammonium phosphate Mono-ammonium phosphate	(Colomba et al., 2007)
$CaCO_3$ with Diammonium phosphate	(Suharty, Almanar et al., 2012)

2.7.1 Inert Flame Retardant Fillers

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Additive of inert fillers can enhance the flame resistance and reduce the smoke produced by polymer.50-60% of non-combustible fillers or inert fillers are added into composite to diluting the mass fraction of organic material. These fillers formed a layer of insulator coating on the surface of composite therefore heat conducted to composite is reduced. By doing this, decomposition of polymer being slowed down and degradation processes happen in longer time (Gibson and Mouritz, 2006).

To become inert fillers, high degree of thermal insulation is major requirement. Calcium carbonate silica or carbon black is suitable as inert filler to increase the flame resistance. (Gibson and Mouritz, 2006).

2.7.2 Active Fire Retardant Fillers

Active fillers always have higher efficiency than inert fillers by acting as a heat sink for composites. When composite decomposed at high temperature, heat is absorbing under endothermic process. At this time, active fillers will absorb the heat prior before the composite does. Hence the temperature is reduced and lowers the rate of decomposition. However, fillers being chosen must has a higher decompose temperature than composite.

In the case of this research, polypropylene decompose at about 3-400^{o}C, and therefore fillers added should has a higher decompose temperature than polypropylene while lower than pyrolysis temperature of 450^{o}C (Gibson and Mouritz, 2006).

2.7.3 Fire Retardant Organic Polymer

There is another effective way to increase fire retardant properties of composite, insert chlorine, bromine, phosphorus into the molecular structure of a polymer or known as

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graft copolymerization. Graft copolymerization is the famous method to implement fire retardant polymer. It enhances the flame retardancy and improving the compatibility of polymer with additive flame retardants (Zhang and Horrocks, 2003).

2.8 Flammability of Composites

Natural fibre composites subjected to thermal decomposition when exposing in fire or high-intensity heat sources. Same situation goes no different for kenaf-based composites. Ignition of fire is the priority needed to eliminate or least we can do is extend its ignition time. Time of ignition widely depends on the heat intensity, oxygen density and air flow in that area. When the fire somehow ignited, works must be done to reduce the fire propagation rate. Fire propagation test is the method used to experiment the rate of fire spread of sample. Lastly, to inspect a fire from overview, several methods were hired, Thermo Gravimetric Analysis (TGA), Different Scanning Calorimetric (DSC) and Dynamic Mechanical Analysis (DMA)

Kenaf fibres are plant base natural fibre. Thus, it is highly flammable as wood. In general, implementation of fire retardants filler into flammable materials such as kenaf-based composites do increase the fire behavior of sample. Chemical compounds are the most common used fire retardant fillers. They usually come in form of powder and contain 5 to 10% of it inside the sample. Particle size is the main factor to influences its efficiency due to the large surface area of fine particle. Nanocomposite is the new area of able to create highly flame retardancy while having high load withstand (Kandola, 2000)

The ordinary matrix for composites is thermo-set, thermoplastic or biopolymer. To select a polymer, Limiting Oxygen Index (LOI) is the prime consideration. LOI is the elementary density of oxygen in percentage that supports combustion of a polymer.

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Table ***728***7 presented the LOI and heat release rate (HRR) for certain polymer. Polypropylene (PP) is a matrix material that always led the others and its flammability is widely studied (Helwig and Paukszta, 2000;Helwig et al., 2000;Li and He, 2004;Borysiak et al., 2006). A measurement of HRR for PP matrix with four natural fibres reinforced combinations conducted by Kozlowski, 2008. The volume of natural fibres in PP matrix was 30% and it is 5mm thick sample (Kozłowski and Władyka-Przybylak, 2008). Although the presence of natural fibre was brought an earlier ignition, yet the heat released of natural fibre composites was noticeable reduced more than 50% of pure PP matrix.

Table 728. Limiting oxygen index (LOI) and heat release rate (HRR) of some polymer

Polymer	LOI	HRR (kW/m^2) (Heat flux 40kW/m^2)
Polypropylene	18	1509
Polyethylene	18	1408
Polystyrene	18	1101
Poly (methyl methacrylate)	18	665
Polycarbonate	27	429
Poly (vinyl chloride)	42	1755

Adopted from Joseph and Ebdon, 2000.

2.8.1 Ignition time

In a fire incident, slow rate of fire propagation is meant to increase the escape time. Yet ignition time more important because it is the moment of panic when people saw the fire. The longer ignition time, the longer escape valuable season without panic. A fire ignition test was held in laboratory by Izran et al. The sample is made by kenaf core particles with 180^{o}C hot press. Few types of fire retardant filler added into sample tend to get better result such as DAP, MAP and BP® (Izran et al., 2010).

The furnace temperature being increased to 183^{o}C or until the board collapsed. Timer is always counting down for ignition time of each sample. Table ***829***8 was listed out the result of the test. Untreated board only required 50seconds to ignite while BP® is able to extend its ignition period more than double of time to 2 minutes. Besides, BP® only has 8.52% of burnt area and 0.69% of weight loss. BP® shows best fire retardants behavior and this had agreed with the research by Abdul Rashid and Chew (Abdul Rashid and Chew, 1990). Boron is capable to provide protection to postpone the heat transfer (Kozłowski and Władyka-Przybylak, 2008).

Table 829. Result of the test

Sample	Ignition time (s)	Burnt area (%)	Weight loss (%)
Untreated	50	18.43	0.99
DAP-treated	100	15.83	0.97
MAP-treated	100	14.28	0.55
BP®-treated	120	8.52	0.69

Adopted from Izran et al, 2010.

Suharty, (2012), used different combination of kenaf fibre composites to get his ignition time finding. Wasted polypropylene (wPP) dumped by people is the matrix for the combination. Kenaf fibre was first cleaned with ethanol, dried in 40^{o}C oven and grounded into 100 mesh particle sizes. Fire retardant filler candidates were chosen as natural $CaCO_3$ (CC), nano particle $CaCO_3$ (nCC), natrium polyphosphate (NaPP) and DAP. The combination summarizes in Table9. The ignition time for waste pure

PP sample and kenaf-PP composite is 1.6 and 2.16 seconds respectively. Kenaf fibres are proven that able to increase the ignition time for one-third. On the same time, "Sample 8" that adding flame retardant additives of nCC crossed-over with NaPP successful dragging the ignition time to 4.95 seconds. However, presences of fire retardant in composite were resulted in decrease of tensile strength due to reduction of its elasticity. Same finding was reported in previous work for waste PP reinforced with wood or rice husk by adding 20% of magnesium oxide, $Mg(OH)_2$ (Sain et al., 2004).

Table 9. Combination of sample.

Sample	**Ingredients**							
	1	2	3	4	5	6	7	8
wPP	100	80	65	65	65	65	65	65
Kenaf	-	20	15	15	15	15	15	15
CC	-	-	7	-	7	7	-	-
nCC	-	-	-	7	-	-	7	7
DAP	-	-	-	-	13	-	13	-
NaPP100	-	-	-	-	-	13	-	13

Adopted from Suharty, Almanar et al., 2012.

2.8.2 Fire Propagation

Fire propagation capabilities depending on the total heat liberated and fire spread for a fuel (Rogowski, 1970;Tewarson, 1994). Heat was emerged by chemical reactions of combustion process. The rate of fire propagation telling us the flame classification of

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the materials that spread across the surface. It can be categorized into four groups in Table ***1030***10 according to BS476: part 7: 1971.

Table 1030. Flame classification.

Classification	Spread of flame at 1.5mins		Final spread of flame		
	Limit (mm)	Limit for one specimen (mm)	Limit (mm)	Limit for one specimen (mm)	
Class 1	165	165+25	165	165+25	Very low rate
Class 2	215	215+25	455	455+45	Low rate
Class 3	265	265+25	710	710+75	Medium rate
Class 4	Exceeding the limits for class 3				Rapid rate

Adopted from Warringtonfire, 2010.

Izran, 2013 had conducted an experiment for kenaf based plastic to test its rate of fire spread when in fire. Core of kenaf fibre was used as raw material harvested from Lembaga Tembakau Malaysia. To increase the fire behavior, 10% w/w of fire retardants is being used. The result was shown in performance index (Table ***1131***11) that calculated by computer software where used to determine the classification of the material. Index values of 10-25 are class 1 (Hall, 1975).

Table 1131. Fire propagation capabilities of kenaf core particle board.

Material	Performance Index (I)	Classification
Untreated	32.6	Class 2
DAP-treated	15.6	Class 1
MAP-treated	16.8	Class 1
BP®-treated	23.8	Class 1

DAP: Diammonium phosphate, MAP: Monoammonium phosphate, BP®: Mixture of Boric acid, Guanylurea phosphate and Phosphoric acid
Adopted from Izran Kamal, 2009.

From the result above, treated board successfully downgrade the classification by slowing the propagation rate. All treated boards have maximum early heat release between 10-15mins while untreated board was before 10mins. MAP was the first sample to cut down the early maximum heat release, but DAP effectively reduces the second maximum heat release among them.

On the research conducted previously, wasted PP is highly flammable and spreading with 2.15mm/min. However, this situation gets much better to 1.09mm/min when kenaf fibre was added. Furthermore, the rate of fire propagation reduced to half for “Sample 7” and “Sample 8” as compared to kenaf /wPP composites, Sample 2. Unfortunately, things never happen happily all the way down. Bio-composites with phosphoric acid fire retardant released one-tenth more of heat. This is because phosphoric acid reacted with the $CaCO_3$ to yield CO_2 and H_2O that inhibit combustion (Zebarjad et al., 2006).

2.8.3 Combustion

A sustainable combustion process required three main components, fuel, oxygen and heat. Kenaf-based composites acts as fuel in this case, therefore thermal decomposition of kenaf fibre and matrix are the important studies for pyrolysis. TGA allowed showing the mass of sample lost according to temperature (Julkapli and Akil, 2010). Thermal decomposition of kenaf fibre always started by low temperature of hemicelluloses decomposition and followed by a sudden drop of mass causing by pyrolysis of cellulosic. Lignin decomposition is the last component to decomposition at highest temperature.

A previous study of TGA behaviour for kenaf fibre reinforced thermoplastic polyurethane (TPU) composites was done by El-Shekeil (El-Shekeil et al., 2012). Different kenaf fibre loadings sample were fabricated by compression molding from 20% to 50%. All of the sample run though first mass loss at low temperature for reason of expelling the moisture. The main temperature peak for pure TPU is around 363°C and caused by polymertization (Figure). In the finding, data shows that increasing the fibre content decreased the thermal stability. However, TPU/kenaf composites have loss about 80% of weight while pure TPU remain not even 10% of total weight (Figure). The first peak of thermal degradation for 20% kenaf fibre loading is 246 °C and shifted to around 217 °C for 50% fibre content. The trend happen is because the thermal behaviour of kenaf fibre taken prior as increasing its content. First peak of mass drop for kenaf fibre is hemicelluloses degradation and begins on around 200 °C (Azwa et al., 2013).

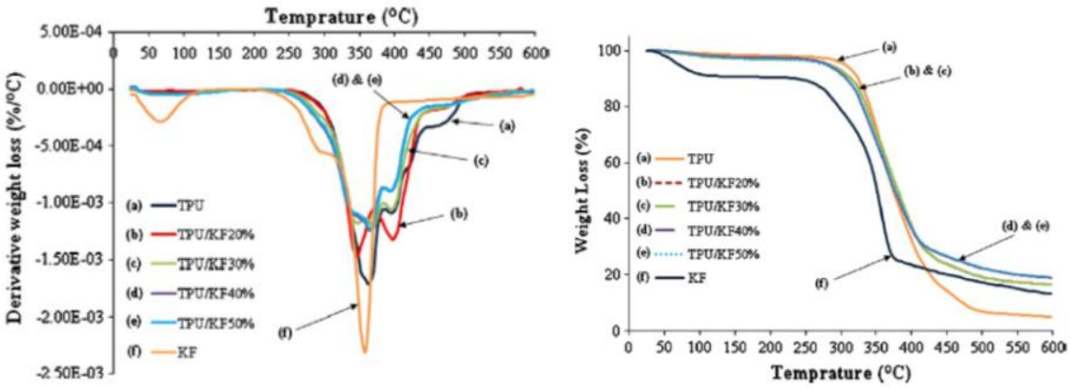

Figure 13. (a) Derivative Thermo Gravimetric (DTG) of TPU/Kenaf composites (b) TGA of TPU/Kenaf composites

(Source: El-Shekeil et al., 2012).

Despites of the factor of fibre content on thermal behaviour, types of matrix used cause variation of result as well. Russo et al conducted an experiment pointing to the several polymers as matrix for kenaf fibre (Russo et al., 2013). Candidates used in research are completely biodegradable poly(3-hyroxybutyrate-*co*-*3*-ohydroxyvalerate) (PHBV), random copolyester of poly(butylenes adipate-*co*-terephthalate) (PBAT) and low-density polyethylene (LDPE).kenaf fibre carried out by both alkalization and silanization. Both PHBV and LDPE matrix based composite had shifted their thermal

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degradation earlier compared to its own pure polymer. The first mass loss for PHBV occurs at around 250^{o}C which caused by chain scission reaction. This temperature is almost close as temperature of degradation of hemicelluloses. Hence polymer PHBV and its composite having same degradation temperature range.

Fire makes things become rupture. Everything in fire will eventually become charcoal, gases and heat. Matrix of the composite will melt away and fibre under thermal decomposition as well. This phenomenon led to reduction of the product’s strength. The DMA program observed the time-dependent deformation behavior x(t) under periodic with very small amplitudes F(t). It is possible to gather information such as Young’s modulus / storage modulus, E’, loss of modulus, E” and mechanical loss factor, tan δ, as a function of deformation (Mazuki et al., 2011). Meanwhile, the DSC could found out some important parameters, like oxidation, glass transition temperature (T_g), crystalline level and melting temperature (Qu et al., 2000).

John et al were conducted an experiment regarding effect of kenaf fibre content in composite when in fire. The composites were prepared from nonwoven kenaf and PP by varying the fibre content from 0% to 40%. Dimension of sample is 50mm x12mm x30mm and testing temperature ranged from -20^{o}C to 150^{o}C. The result found out that storage modulus (E’) increasing by adding kenaf content at all temperature. This is because the increase of stiffness of composite. Besides, increases of loss modulus (E”) when kenaf content is getting higher. Researchers believe it’s caused by increase in energy absorption by the additional kenaf fibres.

Mazuki et al found that is a different pattern of degradation dynamic mechanical properties for pultruded kenaf fibre reinforced composites when pre-treated in different pH solutions (Mazuki et al., 2011). Three samples with 70% of kenaf fibre content were immersed in pH5.5, pH7 and pH8.9 which representing acidic, neutral and alkaline solution respectively for 24 hours. The result showing that the storage modulus of sample was decreased after immersion process. The values were reduced to 4GPa, 3.8GPa and 3GPa for pH7, pH5.5 and pH8.9 separately at their glass transition temperature, T_g (172°C). The peak of tan δ had happened earlier and reduced than standard sample after immersion process. Peak of tan δ is the determination of glass transition temperature, T_g (Kuo et al., 1998). The T_g decrease from 172 °C to 162 °C, 155 °C and 150 °C for neutral, acidic and alkaline solution respectively. These finding values are due to the better ductility after immersion process by restricting the movement of polymer molecules (Rana et al., 1997;Gu, 2009).

Nishino et al had investigated kenaf sheet, PLLA film and its composites under DMA testing (Nishino et al., 2003). Kenaf/PLLA composites shows greater dynamic storage modulus (E') and value maintained up to the melting temperature of 161°C. On the other hands, PLLA film sample showed an unexpected descend in the E' value while kenaf sheet has no changes until 200°C as shown in Figure 53.

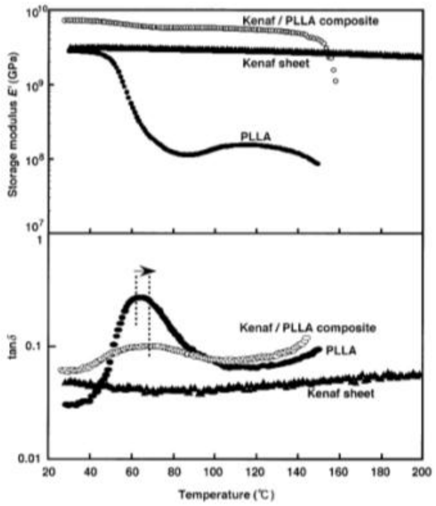

Figure 53. Dynamic storage modulus (E') and mechanical tan δ for kenaf sheet, PLLA film and kenaf/PLLA composites

(Source: Nishino, Hirao et al., 2003).

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Lee, (2009a) investigated the viscoelastic behavior for Kenaf/ PLA composites with DMA test. The composites were pre-treated by silane coupling agent (SCA) in amount of 1 – 5%. They found out the E’ value is getting lower when rising the temperature. This is because of chain fluidity has increased in the matrix. Yet the E’ value is higher by increasing the coupling agent amount throughout the temperature. Besides, He also realized that amount of kenaf content do affect the E’ value of experiment. Percentage of fibre content is directly proportional to its E’ value until 70 wt% of fibre. The high quantity of kenaf fibre restricted the matrix amount. Hence load transfer hardly to be done.

DSC measurement also being conducted in research by Lee (2009). From the result (Table ***1232***12), glass transition temperature, T_g and melting temperature, T_m didn’t influenced by the appearance of silane coupling agent. However, both of heat of fusion (ΔH_f) and degree of crystalline (X_c) had increased about 4J/g and 4% respectively. The incensement of crystalline caused by the kenaf fibre has stimulated into heterogeneous crystallization.

Table 1232: Thermal behavior of kenaf/PLA composites

Sample	**T_g (°C)**	**T_m (°C)**	**ΔH_f(J/g)**	**X_c (%)**
PLA	60.0	160.3	-	-
Kenaf/PLA	56.9	164.2	19.2	20.5
Kenaf/PLA (1% SCA)	58.7	168.6	22.8	24.3
Kenaf/PLA (3% SCA)	59.3	168.1	23.1	24.6
Kenaf/PLA (5% SCA)	59.1	167.3	23.3	24.9

Adopted from Lee et al., 2009a.

In Julkapli & Akil’s research, they perform DSC analysis twice on kenaf-filled chitosan composites (Julkapli and Akil, 2010). First cycle is below 100°C while

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second cycle is below 200°C. The reason to perform first cycle is to eliminate moisture content and thermal stress in the composites because the result highly affected by water in sample (de Britto and Campana-Filho, 2004). One finding result was same as other researchers is the fibre content increase the crystalline degree of the sample. At the same time, increasing the kenaf quantity led to a drop of ΔH_f value.

2.9 Mechanical Properties of Composites

When talking about mechanical properties of certain product, first thinking from mind will relate to strength of the product. Tensile strength is the maximum pulling stress for a material able to withstand before breaking. In Suharty N. S. research, composites have significant high tensile strength of 32.4MPa but some of deteriorate of strength when fire retardant filler put into it (Suharty et al., 2012). This is reasonable due to attendance of filler had lowered elasticity of composite. Some of material requires not only strength but also ability to stand tall under impact. Kenaf fibre reinforced polypropylene composites allowed to absorb double of impact value from what pure polypropylene can works. Synthetic polymers like polypropylenes are not bio-degradable. This means the waste polymer will accumulate every year and create severe landfill issue. The appearances of bio-gradable natural fibre in composites help to increase biodegradation ability. A research proved that non-degradable polypropylene degrades for 5.75% of its weight by adding kenaf fibre.

Apart from this, fibre treatment and fibre content in composites do affect its properties. Asumani, 2012 revealed that 5% of NaOH alkali-silane fibre treated is the optimum portion to create highest tensile strength. The more fibre content in the composites the higher tensile strength (Asumani et al., 2012). This statement is applicable for below 30% of fibre content. Now of more than 30% of fibre content, the tensile strength

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reducing. This is because of imbalance of fibre/matrix ratio, too little of matrix prohibited load transfer evenly.

Different kind of production requires different manufacture method. In fact, manufacture method influences properties of product. Asumani, 2012 and Meon, 2012 carried out separate experiment by use same 30% short NaOH treated kenaf fibre content reinforced with polypropylene and maleic anhydride polypropylene coupling agent. The only difference is Asumani, 2012 using kenaf mat sandwiching between polypropylene sheet and powder by compression moulding while Meon, 2012 mix all the ingredients and chopped with crusher machine before putting into cold press machine. Both results consist of more than 10MPa differences (Asumani, Reid et al., 2012;Meon et al., 2012). Figure 1554 illustrated the result of tensile strength of alkali-silane treated composites and Figure 55 shown the tensile strength of adopted from Meon. Other than this, Bernard and Zampaloni both studied on the effect of manufacture parameters on kenaf fibre reinforced poplypropylene composites (Zampaloni et al., 2007;Bernard et al., 2011).

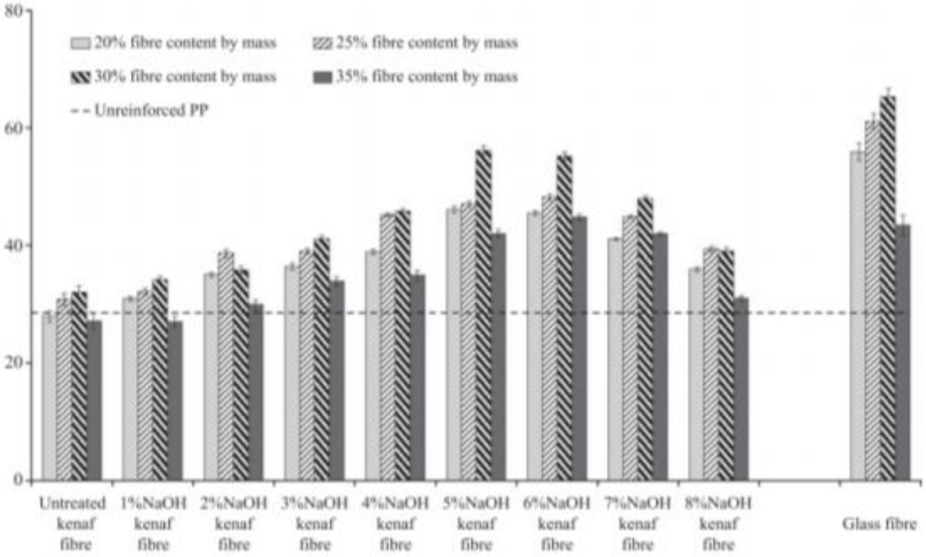

Figure 1554. Result of tensile strength of alkali-silane treated composites.
(Source: Asumani, Reid et al., 2012)

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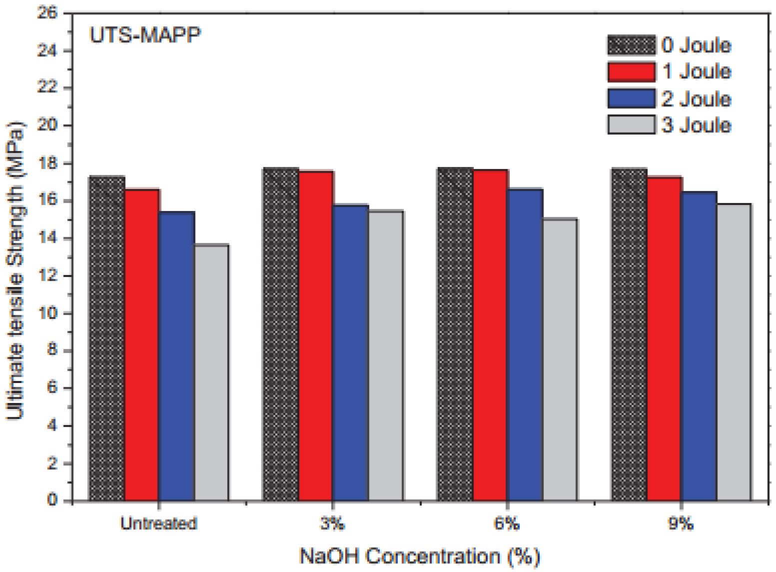

Figure 55. The tensile strength of alkali-silane treated composites.

(Source: Meon et al., 2012)

2.10 Discussion

Phenolic resin with either fibreglass or graphite is the composites used for aircraft interior panel fabrication. High fire-retardant properties of phenolic resin postponed the ignition time and hence extend the escape time frame for survivals. However, environmental issues getting more concerned by society. Thus, natural fibres and biodegradable resins were being used and replaced. Kenaf fibres reinforced Floreon resins have been chosen in this research. Floreon has been proven high strength, light weight and low in cost that shall apply in many applications such as door panel. Most importantly, Floreon composite is environmental friendly.

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CHAPTER 3

METHODOLOGY

Methodology is a very important step to narrow down the factor of influences of the data collected. Methodology also helps to ensure the direction of research will be in accordance to the objectives. In this methodology chapter, the material used, sample preparation route, flow chart will be discussed.

3.1 Materials

The PP used was Propelinas 600G (homopolymer), which was purchased from Polypropylene (M) Sdn. Bhd. with a melt flow index of 12.00 g/10min. Meanwhile, the biopolymer used was Floreon (FLO) resin Grade 100, which was supplied by The University of Sheffield. KF originated from Malaysia and their lengths ranged from 8-15mm and were measured by suppler while MH with 95% of purity was used to improve flame retardant behaviour of composites. Both KF and MH were supplied by by Tazdiq Engineering, Serdang, Malaysia. Furthermore, the maleated polypropylene (MAPP) based coupling agent (E-43) was used has a density of 0.930 g/cm^3, molecular weight of 9100 g/mol and supplied by Suka Chemicals (M) Sdn. Bhd. Besides, Magnesium hydroxide (MH) which was purchased from Fisher Scientific UK Ltd while the sodium hydroxide (NaOH) used in the alkali treatment was contributed by APC Pure, UK.

3.2 Composite Preparation

Table ***1635***13 lists the different ratios of FLO and PP composite samples, which were prepared using a 21mm lab twin screw extruder. Here, KFs were dried for 24 hours at 50 ^{o}C before treated with 6 % NaOH, for 4 hours. Then, it was washed and rinsedby using water and dried for 6 hours at 100°C. The working temperature for the extrusion during compounding was set at 180 °C at the die head and was increased to 186 °C at the feed section with 50RPM while the PP composite was using 190 °C at the die head and increased to 200°C. The process method was adapted from a previous study (Zhang et al., 2012). The extruded strands were then dried by blowing air before being pelletised. The pellets were then carried to a hot press machine in The University of Sheffield Csic laboratory for sheet fabrication. The setting of the hot press machine was set to five bar of pressing pressure for 10 minutes, before going through 10 minutes of pre-heating. The mould was then allowed to cool for 10 minutes in room temperature.

Table 1333:Combination formula of KF/FLO/MH composites.

Sample name	Floreon (FLO), wt%	Kenaf fibre (KF), wt%	Magnesium Hydroxide (MH), wt%
Pure FLO	100	-	-
5KF	95	5	-
10KF	90	10	-
5MH	95	-	5
10MH	90	-	10
5KF5MH	90	5	5
5KF10MH	85	5	10
10KF5MH	85	10	5
10KF10MH	80	10	10

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3.3 Characterization Techniques

In the meantime, TGA testing was conducted under a nitrogen atmosphere in order to determine the thermal stability of the composites. Samples of 5 mg were prepared for the testing. The samples were heated from ambient temperature to 600 ^{o}Cwith a heating rate of 20 ^{o}C/min. The results gained were then analysed by computer software to extract the onset temperature, the 2nd peak, mass loss and mass residual.

3.3.1 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) was used to study the morphology of the specimen where the cross-sections of flexural testing specimen represent the location of study. The accelerating voltage of 15kV helped to image the cross-sections.

3.3.2 Tensile Testing and Flexural Testing

Tensile tests were carried out with ASTM D638-14 using the Instron 5kN machine locate in Universiti Putra Malaysia (UPM) (2014). A One mm thick dumbbell specimen was cut from the moulded sheet while a cross-head speed of 1mm/min was used and the test was performed at 25 ^{o}C. Flexural testing was conducted using the same Instron 5kN machine in UPM according to ASTM D790-10 testing standard (Venkateshwaran and Elayaperumal, 2010). The dimension of the specimen was 96 mm × 12.7 mm × 5mm, which included 10% of the support span for overhanging.

3.3.3 Impact Testing

The un-notched specimens were subjected to Charpy impact testing which adheres to ASTM D6110-10 (2010). The dimension of the test specimen was 127 mm × 12.7 mm × 5 mm. Each specimen was measured with Verniercaliper before the impact testing was conducted where the accuracy of the reading was up to 0.1 mm. The specimen was then positioned horizontally on the supports of the impact testing machine. The pendulum was raised and secured in the release mechanism and the indicating board was reset. The pendulum was then released to provide impacts to the specimen; consequently, the indicated readings were recorded for impact strength calculation.

3.3.4 Water Absorption testing

Test specimen was initially dried at 50 °C for 24 hours by following ASTM D570-98(2010)e1 (2010). The specimens were then placed so that they were entirely immersed in a container of distilled waterwhich was prepared by the UPM chemical laboratory. At the end of the first, second and 24th hours, the specimens were removed from the water and all of their surfaces were wiped off with a dry cloth. Here, the weighing scale was nearest to 0.001g and one specimen was put back into the container before the next one was taken out andthe process was continued until all the specimens had been processed. After that, these drying and weighing procedures were repeated for every 24 hours until day 7. Then, the repeating duration was changed to every weekly (7 days) until all specimens had been saturated. In this light, each specimen was considered as saturated when only less than 1 % of its total weight (5mg) increased. In all, five specimens were tested for each group, and average results were recorded and reported.

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3.3.5 Thermogravimetry Analysis (TGA)

TGA measurement was performed under nitrogen flow from ambient temperature to 600 ^{o}C by a rate of 30 ^{o}C/min in Chemical Engineering Department at Universiti Putra Malaysia (UPM). The weight of the sample used was roughly 10 mg and performed by technician in Chemical Engineering Department.

3.3.6 Differential Scanning Calorimetry (DSC)

DSC measurement was performed at the Csic laboratory in University of Sheffield with a Perkin Elmer DSC 8000 Advanced Double-Furnace. The crystallization temperature (T_c), the melting temperature (T_m) and the glass transition temperature (T_g) were evaluated in the testing. Approximately 5 mg of the samples were sealed in an aluminium plate. The testing was started at ambient temperature and raised up to 200 ^{o}Cat a rate of 10 ^{o}C/min (Sacchetin et al., 2016). The sample was held at 200 ^{o}C for a minute and then its temperature was decreased back to ambient temperature with a 10 ^{o}C/min cooling rate. The sample was held at ambient temperature for a minute and immediately a 2nd heating cycle was conducted up to 200 ^{o}C with same heating rate. The first heating cycle was used to remove the thermal history and moisture from the sample.

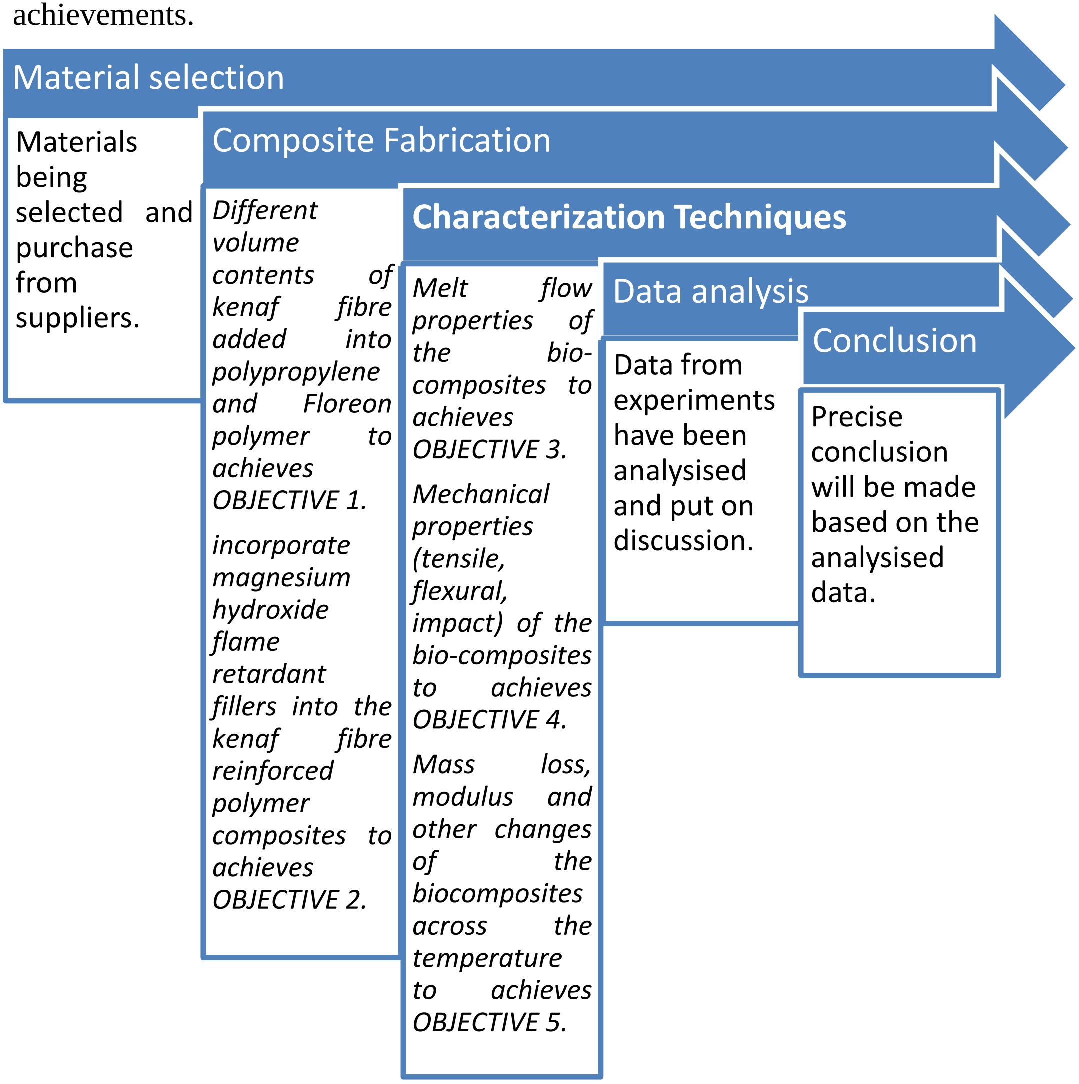
3.3.7 Dynamic Mechanical Analysis (DMA)

DMA testing under a dual cantilever mode was implemented with a sample dimension of 50 mm x 10 mm x 5 mm. The measurements were carried out in the temperature range from ambient temperature up to 200^{o}C, with a scanning rate of 2^{o}C/min at a frequency of 1Hz. The sample was clamped on both sides and the stress, σ was applied sinusoidally by a movable clamp at the centre of the clamped sample. The response from the sample was measured as strain, ε. The strain was obtained as the retarded lag of the stress applied due to the behaviour of the sample. The dynamic modulus, E', and the dynamic loss modulus, E'', were determined by the phase angle, δ, which was calculated from Eq. 2:

$\tan\delta = E''/E'$ Equation 2 (Idicula et al., 2005)

3.4 Flow Chart of the thesis

The figure 17 shows the flow chart of this study accordance to objective achievements.



Material selection

Materials being selected and purchase from suppliers.

Composite Fabrication

Different volume contents of kenaf fibre added into polypropylene and Floreon polymer to achieves OBJECTIVE 1.

incorporate magnesium hydroxide flame retardant fillers into the kenaf fibre reinforced polymer composites to achieves OBJECTIVE 2.

Characterization Techniques

Melt flow properties of the bio-composites to achieves OBJECTIVE 3.

Mechanical properties (tensile, flexural, impact) of the bio-composites to achieves OBJECTIVE 4.

Mass loss, modulus and other changes of the biocomposites across the temperature to achieves OBJECTIVE 5.

Data analysis

Data from experiments have been analysised and put on discussion.

Conclusion

Precise conclusion will be made based on the analysised data.

Figure 56. Flow Chart

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CHAPTER 4

MECHANICAL AND THERMAL PROPERTIES OF KENAFFIBRE REINFORCED POLYPROPYLENE/ MAGNESIUM HYDROXIDE COMPOSITES

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ABSTRACT

This paper presents a study of the mechanical and thermal properties of kenaffibre (KF) reinforced polypropylene (PP)/magnesium hydroxide (MH) composites. Pure PP sample, has shown low tensile, flexural and flame retardant properties. It was found that KF and MH filler insertion had improved the properties of PP composites. The increment of KF contents in composites had shown higher tensile modulus and decomposed mass loss at onset temperature, butlower values in tensile strength, elongation, flexural strength and onset temperature. In the meantime, 25 wt% KF contented composite shown a slightly higher flexural strength, while the higher volume of MH filler in composites caused lower strength, tensile modulus, elongation, but higher onset temperature and the 2nd peak temperature in thermogravimetric analysis (TGA) testing. Furthermore, increasing the KF contents in PP matrix has found lower mass residue. However, increasing of KF contents in MH contented composite had increased the mass residue at the end of the testing.

Keywords: Kenaffibre; Polypropylene; Magnesium Hydroxide; Biocomposites; Mechanical properties; Thermogravimetric analysis

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NOMENCLATURE

KF	Kenaffibre
PP	Polypropylene
MH	Magnesium hydroxide
TGA	Thermogravimetric analysis
RPM	Revolutions per minute

INTRODUCTION

In recent years, environmental issues have become important in every technological sector, especially in advanced materials. Outstanding performance, along with environmentally friendly materialshave gradually substituted conventional materials. As a result, naturalfibres have been widely chosen in the reinforcement of polymer composites to achieve the designated properties (Sastra et al., 2006;Maleque et al., 2007;Leman et al., 2008;Leman et al., 2008;Anwar et al., 2009;Rashdi et al., 2009;Zainudin et al., 2009;Cerqueira et al., 2011;Yang et al., 2011;Ishak et al., 2013;Yahaya et al., 2015). Nowadays, natural fibre reinforced polymer composites have been extended to advanced applications, such as in automotive, aircraft, medical and food industries (Giancaspro et al., 2009;Thakur and Thakur, 2014). Many types of natural fibrehave also been used to reinforce PP polymer, e.g. flax fibre (Ausias et al., 2013;Doumbia et al., 2015;Wang et al., 2015), hemp fibre (Rachini et al., 2012;Etaati et al., 2013;Yan et al., 2013;Etaati et al., 2014;Panaitescu et al., 2015) and jute fibre (Aggarwal et al., 2013;George et al., 2013;Karaduman et al., 2014;Yallew et al., 2014).

KF/PP composites were found to demonstrate outstanding flexural properties among other natural fibre/PP composites (Bledzki et al., 2015). High cellulose and good orientation of microfibrills in KF are the main reasons for KF/PP composites to have high flexural properties. The reinforcement fibrehas changed the composites from

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elastic to tough and rigid properties, where high stiffness and strength had been found. Besides that, a shift to the higher crystallization temperature had been achieved in KF/PP composites and thisis related to the high crystalline content of KF. KF/PP composites also showed higher storage modulus, E' throughout the temperature due to the high energy absorption ability of KF. This has led to a higher stiffness behavior for KF/PP composites (John et al., 2010). However, the introduction of KF in PP matrix has caused a reduction in the peak thermal degradation temperature (Lee et al., 2013). This is attributed to the lower thermal stability of KF compared to PP matrix (Lee et al., 2014). The rapid degradation of KF has accelerated the thermal degradation of neighboring component.

To enhance the fire properties of natural fibre reinforced polymer composites, the inclusion of flame retardant filler is one of the effective solutions. The incorporation of flame retardant can disrupt the burning process by formatting a foam multi-cellular char on polymer surface or emitting water vapor to dilute the fuel concentration at the surrounding (Ismail et al., 2013). In this light, MH flame retardant is one of the most popular flame retardants to enhance the thermal properties of composites. Consequently, MH is drawing much public attention due to its high decomposition temperature and its smoke suppressing ability (Liu, 2014); its alkaline nature is useful to naturalize the acidic gases (NO_x, SO_2, and CO_2) (Ren et al., 2013). Therefore, it is considered as an environmentally friendly flame retardant. Sain *et al.* (2004) conducted a study of the effect of MH on properties of rice husk/PP composites and sawdust/PP composites whereA 25 wt% of MH content in the composites resulted in 50%reduction of flammability. Meanwhile, Stark *et al.* (2010) had found similar improvement in their studies and MH had shown the best fire retardant effect among five flame retardants in wood/PP composites. Another study showed the improvement of results of thermal stability of MH/oil palm empty fruit bunch fibre/PP composites and these can clearly be seen from higher decomposition temperature located in TGA (Ridzuan et al., 2013). Furthermore, high thermal resistance and heat capacity of MH flame retardant were the reasons for MH insertion. On the other hand, sisal fibre/MH/PP composites shown higher char residue compared to the sisal fibre/PP composites (Suppakarn and Jarukumjorn, 2009). In this regard, the formation of magnesium oxide on the composite's surface at high temperature had protected it from further burning. The water vapour, a side product of magnesium oxide formation had diluted the concentration of fuel, hence, slowing down the burning process (Suppakarn and Jarukumjorn, 2009).

From the above review, it is evident that were no previous work conducted on the mechanical and thermal properties of KF reinforced PP composites with MH, which are used as flame retardant filler. Therefore, the aim of this present work is to study the effects of MH inclusion on mechanical and thermal properties of KF reinforced PP composites.

EXPERIMENTAL

Materials

The PP used was Propelinas 600G (homopolymer), which was purchased from Polypropylene (M) Sdn. Bhd. with a melt flow index of 12.00 g/10min.Meanwhile, KF originated from Malaysia and their lengths ranged from 8-15mm and were measured by suppler while MH with 95% of purity was used to improve flame retardant behaviour of composites. Both KF and MH were supplied by byTazdiq Engineering, Serdang, Malaysia. Furthermore, the maleated polypropylene (MAPP)

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based coupling agent (E-43) was used has a density of 0.930 g/cm^3, molecular weight of 9100 g/mol and supplied by Suka Chemicals (M) Sdn. Bhd.

Composite Preparation

The composites consisted of PP, KF and the flame retardant MH. 3 wt% of the MAPP coupling agent was added into every composites combination to improve the surface interaction. At the same time, pure PP was applied as a control sample and the composition of the sample is summarised in With the increase of the KF contents, the poor compactness of the composite caused weaker bonding strength between the matrix and the fibre. Hence, a decline of the tensile strength was found (Tian et al., 2015). In addition, high fibre contents in the composites had induced insufficient number of resin for fibre wetting (Anbukarasi and Kalaiselvam, 2015). In this light, bad wetting affects the load transfer mechanism in the composite, leading to low strength properties. The lack of resin also made it hard to maintain its structure. In this case, the sample failed before it could reach the highest absorbable strength of the fibre.

. All of the materials were dried in the temperature of over 50^{o}C for 24 hours while melt-mixing was performed by HaakeRheocordin the mtemperature of 170 ^{o}C at 50 revolutions per minute (RPM) for a duration of 15 minutes. PP and KF were melt blended with MAPP and MH using the same internal mixer. In this light, the PP was first inserted and melted for 5 min, then the fibres, MAPP and MH were added into the melted PP matrix with correct portion for another 10 min. The mixture was then taken out from the mixer while it was still hot to prevent it from solidifying. After that, the mixture was put into a 1 mm and 3 mm square mould for compression using the Scientific Laboratory Hydraulic Press Type LP-S-80. The parameter of pre-heating

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and final compression sections in the compression were set at 200 ^{o}C for 5 minutes. After that, the compressed sheet was cooled down for 5 minutes in 50 ^{o}C. In all, 16 repeated fabrications processing with different combinations were conducted.

Characterization Techniques

Tensile tests were carried out with ASTM D638, using Instron 5kN machine located in Universiti Putra Malaysia (UPM) (D638-14, 2014). A 3.1 mm x 9.5 mm neck section in63.5 mm x 9.5 mm x 1 mm dumbbell specimen was cut out from the molded sheets by using a die. A cross-head speed of 1mm/min was also used. The tests were performed at 25 ^{o}C. Flexural testing was also conducted in the Instron 5kN machine at UPM by following the ASTM D790 testing standard (D790-10, 2010). The dimension of the specimen was 60 mm x 10 mm x 3mm, including overhanging on each end, which was at least 10% of the support span. In this process, 10 samples were tested in each testing.

In the meantime, TGA testing was conducted under a nitrogen atmosphere to determine the thermal stability of the composites. Samples of 5 mg were prepared for the testing. The samples were heated from ambient temperature to 600 ^{o}Cwith a heating rate of 20 ^{o}C/min. The results gained were then analysed by computer software to extract the onset temperature, the 2nd peak, mass loss and mass residual.

RESULTS AND DISCUSSION

Tensile Properties

Figure 57 and Figure show the tensile strength and tensile modulus for KF reinforced PP composites with MH fillers. Here, the pure PP resin acts as a control sample. It was found that all samples have higher tensile properties than pure PP, other than elongation at break. This is because the KF and MH filler were used as the load bearing in the composites. The 10KF sample had the highest tensile strength of 39.80MPa and tensile modulus of 1143 MPa. However, their tensile strength dropped by 30.90 % from 10KF to 25KF. The tensile strength value wasfound to decrease when the contents of KF or MH increased. Besides that, further deterioration of strength was found when the composite simultaneously increased the KF and MH contents.

Table 14. Specimens' composition and thermogravimetric analysis (TGA) data.

Specimens	KF fibre (%)	MH filler (%)	Onset temperature (°C)	Mass loss (%)	2nd peak (°C)	Mass residual (%)
Pure PP	-	-	383.0	99.79	-	0.210
10KF	10	-	318.8	8.497	441.6	2.324
15KF	15	-	318.3	9.664	442.6	2.911
20KF	20	-	313.3	10.03	442.0	2.819
25KF	25	-	322.9	24.09	395.2	0.648
10KF10MH	10	10	317.6	8.115	460.2	8.118
15KF10MH	15	10	310.0	10.42	460.0	8.279
20KF10MH	20	10	313.5	11.95	461.0	9.019
25KF10MH	25	10	306.2	15.00	461.5	9.407
10KF15MH	10	15	315.7	8.922	463.1	10.45
15KF15MH	15	15	305.1	11.96	461.3	11.12
20KF15MH	20	15	308.2	11.70	460.4	12.71
25KF15MH	25	15	301.3	17.91	458.3	13.81
10KF20MH	10	20	331.6	7.810	464.0	13.80
15KF20MH	15	20	328.8	9.866	461.7	14.56
20KF20MH	20	20	309.8	14.80	460.5	14.75
25KF20MH	25	20	295.9	17.03	457.8	16.31

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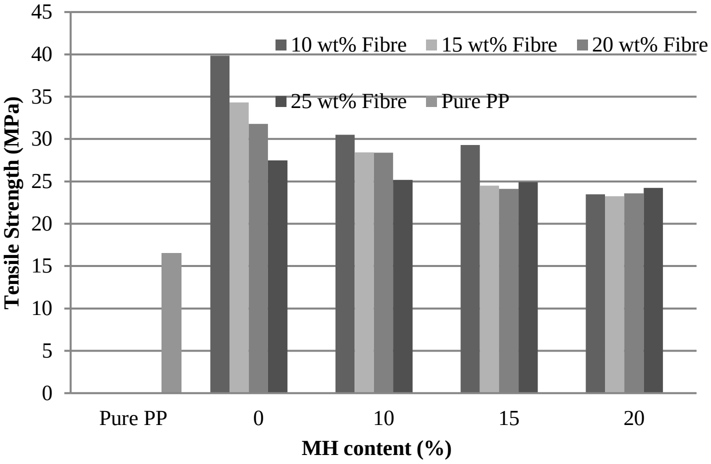

Figure 57. Tensile strength for KF reinforced PP composites with MH fillers.

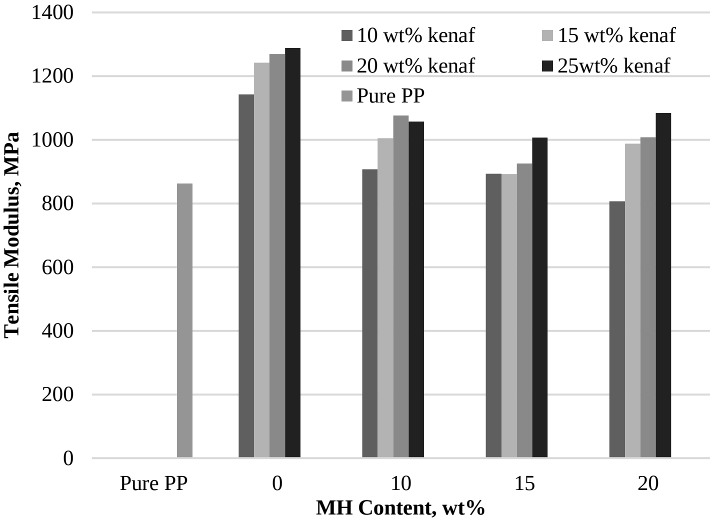

Figure 19. Tensile modulus for the KF reinforced PP composites with MH fillers.

With the increase of the KF contents, the poor compactness of the composite caused weaker bonding strength between the matrix and the fibre. Hence, a decline of the tensile strength was found (Tian et al., 2015). In addition, high fibre contents in the composites had induced insufficient number of resin for fibre wetting (Anbukarasi and Kalaiselvam, 2015). In this light, bad wetting affects the load transfer mechanism in the composite, leading to low strength properties. The lack of resin also made it hard to maintain its structure. In this case, the sample failed before it could reach the highest absorbable strength of the fibre.

The MH flame retardant further decreased the strength of composites. Consequently, theinsertion of flame retardants had a negative impact on the tensile properties. The greatest drop (23.45 %) in tensile strength was found for the appearance of MH in composites, from the 10KF to the 10KF10MH composite specimen. However, the deterioration of strength was insignificant after further addition of the MH flame retardant filler (15 and 20 wt%). The deterioration of the composites'mechanical properties for composites with the flame retardants was also reported (Datta and Kopczyńska, 2015).

Figure 58 shows the elongation of KF reinforced PP composites with MH fillers. The elongation on breakwas dramatically reduced when the KF and/or MH fillers were inserted. It was predictedthat filler insertion will cause the sample to be more rigid. There was non-linear slight decrease in the elongation when the MH content in composite was decreased. Consequently, Only 0.089% of decrement from 0MH to 20MH for 25% fibre contented sample. Comparable results have found in previous research (Liu et al., 2009;Balakrishnan et al., 2012;Liu, 2014). On the other hand, reduced in elongation has been recorded by adding KF content into composites. The KF insertion made the composites to be more rigid and the results found are aligned with previous studies on Alfa fibre reinforced PP composites and KF reinforced PP composites (Keller et al., 2000;Amran et al., 2015;El-Abbassi et al., 2015).

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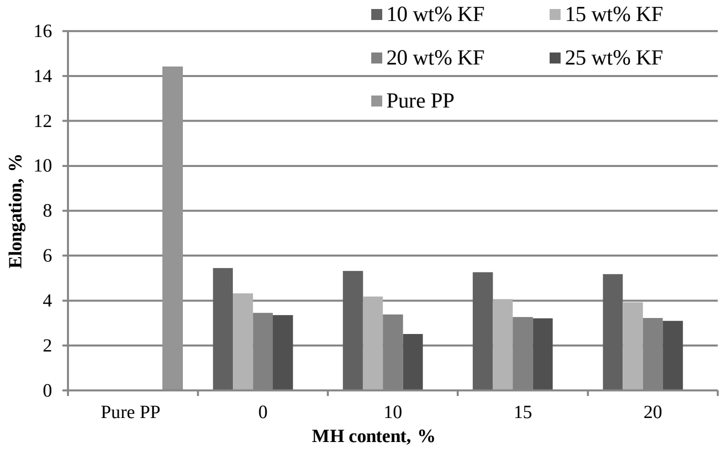

Figure 58. Elongation of KF reinforced PP composites with MH fillers.

Flexural Properties

Figure 59 and Figure show the flexural strength and flexural modulus for KF reinforced PP composites with MH fillers. It was found that all samples have higher flexural properties than pure PP. This is because the insertion of fibre and filler had enhanced its strength. However, the flexural strength had dropped by 14.79 % from 10KF to 20KF, yet, the 25 wt% KF contented composite have a slightly higher flexural strength. This is similar to what observed in a previous study (MdRadzi et al., 2015). All KF reinforced PP composites had shown similar trending for all kinds of MH content (0-20 wt%), except for 25KF20MH sample, which has a flexural strength lower than 20KF20MH. It is believed that high content of KF and MH filler causing insufficient matrix content in the composite.

There are some factors that led to the reduction of the flexural strength. As the fibre content increased the number of fibre ends had increased simultaneously. It actedas a stress concentration spot and the crack began at fibre ends, leading to the flexural strength reduction (Carrot et al., 2012). On the other hand, one must take into account that strength reduction was due to the damage of the fibres during processing at high temperature (Vijaya Ramnath et al., 2013). The higher the content of the fibre in composites, the greater the damage to the composite properties.

The agglomeration of the MH fillers could be another factor responsible for reduction flexural properties. These agglomerates were observed at short mixing times. Its highly hydrophilic behaviour attracted the MH themselves with a strong bonding. The inconsistencies of material dispersion in the composite turned agglomeration spots into concentration points. Carrot *et al.* (2012) explained that in some areas, the large amount of agglomerated MH fillers in an olefinic polymer was due to the low viscosity of the melt and the polymer matrix was unable to break the agglomeration for small particle fillers like MH. Figure shows the dispersion of the MH (in black) in the polymer matrix (in white) by scanning electron micrograph (SEM) image, showing agglomeration of MH even in low volume content.

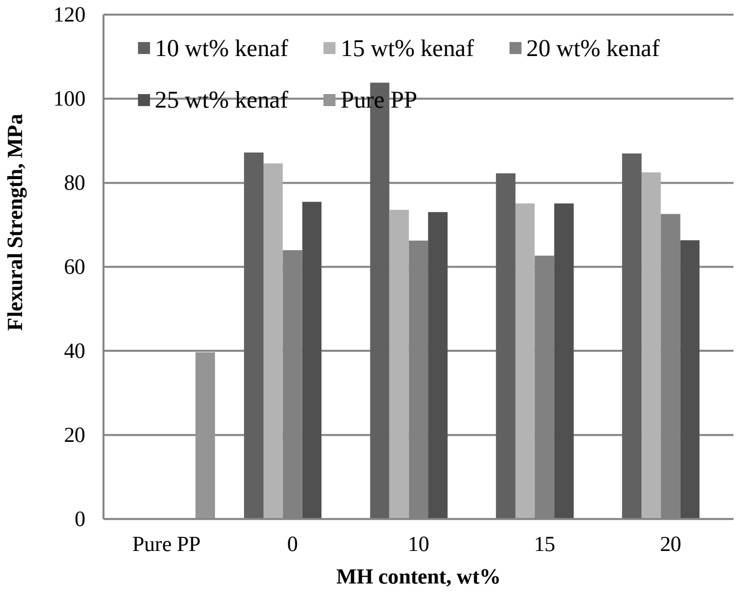

Figure 59. Flexural strength for KF reinforced PP composites with MH fillers.

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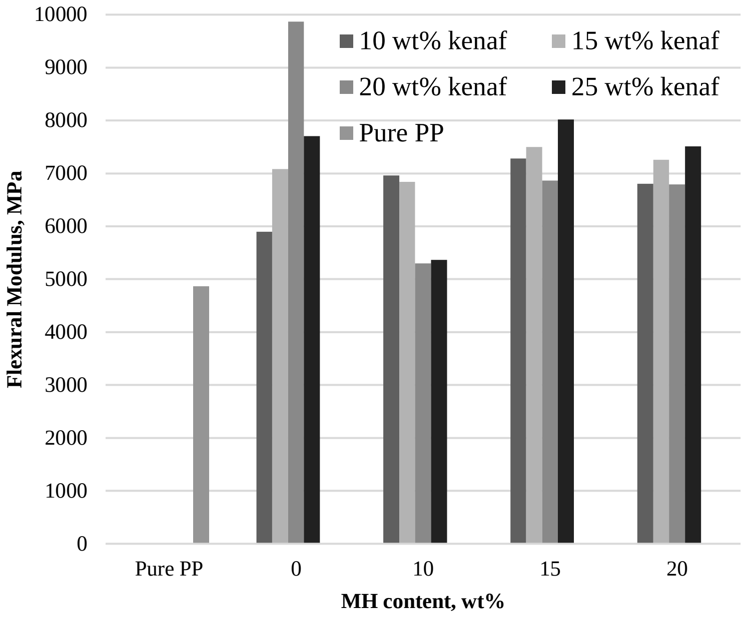


Figure 22. Flexural modulus for KF reinforced PP composites with MH fillers.

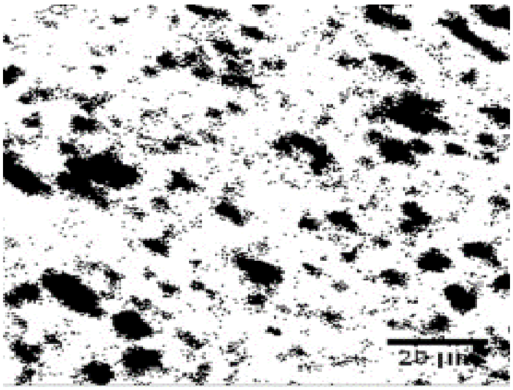


Figure 23. Scanning electron micrograph (SEM) image of dispersion of the MH (in black) in the polymer matrix (in white).

(Source: Carrot, Olalla et al., 2012).

The flexural modulus showed non-linear increases for the KF reinforced PP composite without insertion of MH filler until 20KF, suggesting that the optimum fibre volume on flexural modulus was 20 wt% for composites without MH fillers. On the other hand, the highest flexural modulus in MH contented composites group was 15 wt% of MH. Ismail *et al.* (2013) agreed with these results where the flexural modulus has increased when flame retardant content increased from 20 wt% to 30 wt% due to the improved dispersion of flame retardant in composite. Lastly, a slight reduction of modulus values from 15 to 20 wt% MH contented composites group was also reported. It is believed that high content of KF and MH filler causing insufficient matrix content in composite.

Thermogravimetric Analysis (TGA)

Figure 60 show the TGA for all sample composites. The testing was run under N_2 atmosphere and this parameter was used by previous works to study the flammability behavior of MH on polymer composites (Shehata, 2004;Liu, 2014;Pilarska et al., 2014). The thermal degradation process of a composite started with the moisture weight loss. The moisture attached on the natural fibre was first evaporated at around 100 ^oC.

All PP polymers had decomposed at 383^oC, and contained only one-stage thermal decompositions. This indicates that the PP polymer had demonstrated the thermal retardant properties. In the meantime, the insertion of fibre and filler put the thermal decomposition into two stages, first, a shift to lower temperature and another, a shift to higher temperature. All composite samples had higher mass residual compared to pure PP polymer. However, the higher mass loss was found when the fibre content in the composite was increased. This suggests that by increasing the fibre contents, the cellulose and hemicellulose substances in the composites gradually increased and causing higher mass loss at the onset temperature. Therefore, the higher mass loss values for the higher fibre contents composite. Cellulose and hemicellulose degradation were attributed to the mass loss on the onset temperature corresponded (Gašparovič et al., 2009;Balakrishnan et al., 2012). The 10KF sample has a mass loss of 8.497% and this increase to 9.664%, 10.03% and 24.09% for 15KF, 20KF and 25KF sample respectively. On the other hand, KF has worse thermal stability than PP matrices and therefore, the higher finer contents had induced a lower onset temperature and higher mass lost at the onset temperature.

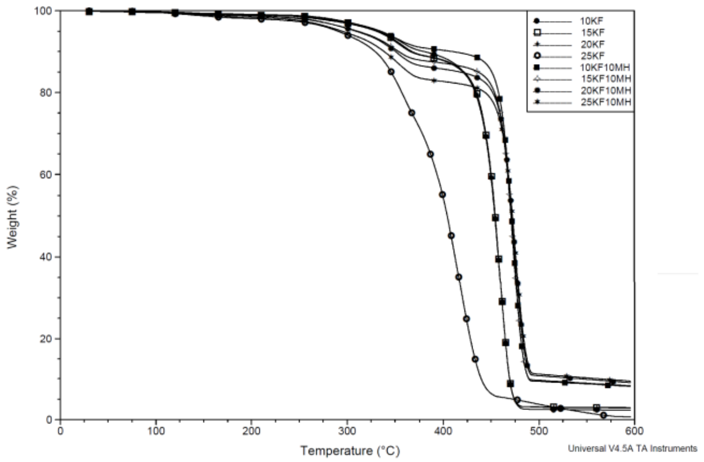
The inclusion of the flame retardant filler MH was meant to improve the thermal stability (higher onset temperature). However, the effectiveness of MH fillers in enhancing the thermal stability of composites depended on the KF loading. Here, KF threshold loadings were 20, 20 and 15 wt% for 10, 15 and 20 MH wt% loading. As a result, the amount of MH fillers was unable to counteract the flammability brought by KF. Moreover, further addition of KF loading continued to reduce the thermal stability by decreasing the onset temperature. Therefore, a higher loading of MH filler will be considered in future development.

Increasing of KF contents in the MH contented composite had also increased the mass residue at the end of the testing. However, an increasing trend was found by increasing the KF contents in PP matrix composite, which also shown lower mass residue. This might be because the MH was protecting the lower thermal stability KF from being decomposed.

On the other hand, Shen *et al.* (2009) found that the results obtained from air atmosphere were slightly different in N_2 atmosphere and the decomposition temperature of MH did not change in both atmospheres. However, the reduced effect of physical barrier of inorganic filler was found under N_2 atmosphere. Therefore, further investigation TGA should be done under air atmosphere.

CONCLUSIONS

The pure PP sample which is the control sample had shown low tensile, flexural and flame retardant properties. It was evident that the insertions of KF and MH filler have improved the properties of PP composites. However, the insertion of KF into the MH contented sample composite found a lower tensile strength, due to the poor compactness between them. On the other hand, low interfacial strength, insufficient polymer content on fibre wetting and higher amount of stress concentration spots had caused the lower tensile and flexural properties. Besides that, the increasing content of KF had shown lower thermal stability. Fortunately, the flame retardant MH filler did enhance the flame retardant properties of composites by showing a higher peak temperature and a lower mass loss. However, the MH content in this study was unable to counteract the flammability brought by the high volume of KF. Therefore, higher loading of MH filler will be considered in future development. Lastly, a higher mass residual was observed at the end of TGA experiment for KF/PP/MH composite. This is because the MH was absorbing the heat and protected the fibre from being decomposed.



(a)

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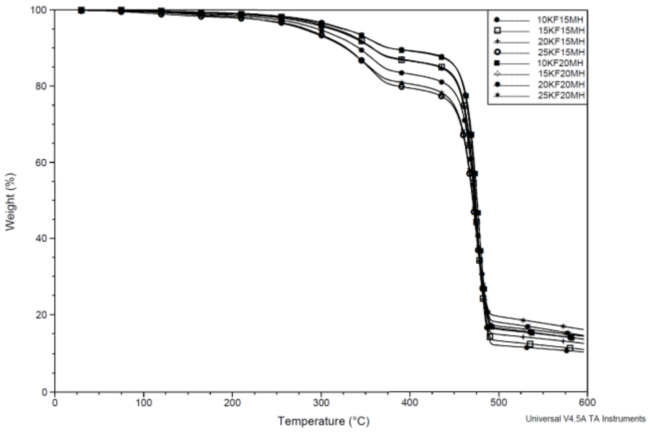

(b)

Figure 60: TGA for (a) 10KF,15KF,20KF,25KF,10KF10MH,15KF10MH, 20KF10MH ,25KF10MH(b) 10KF15MH,15KF15MH, 20KF15MH ,25KF15MH,10KF20MH,15KF20MH, 20KF20MH ,25KF20MH.

ACKNOWLEDGMENTS

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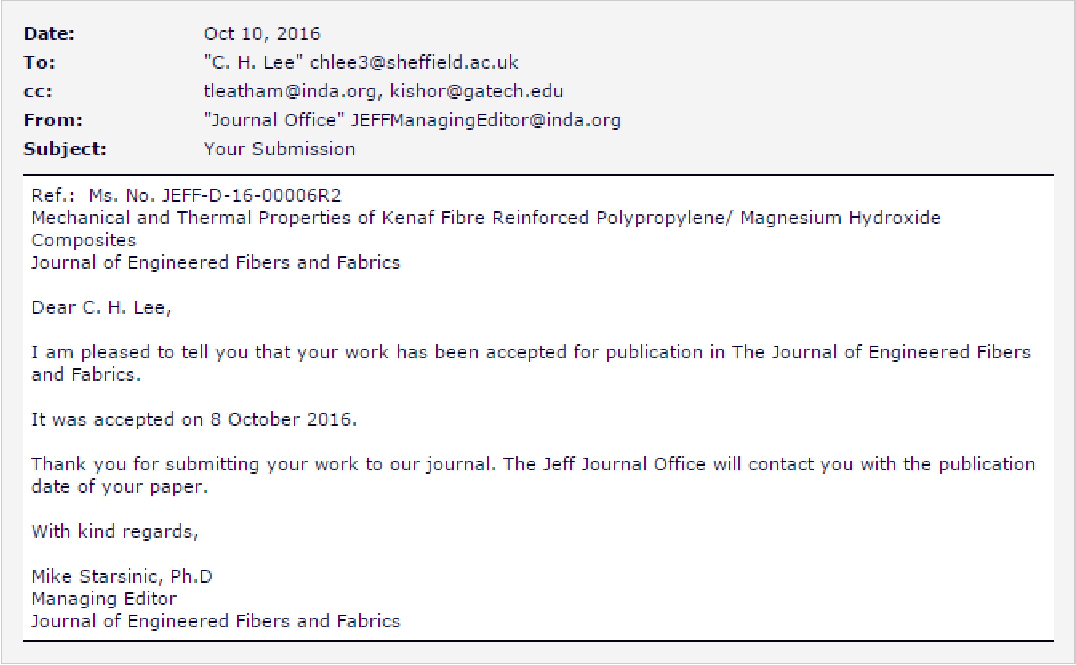
Acceptance email



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Ref.: Ms. No. JEFF-D-16-00006R2
Mechanical and Thermal Properties of Kenaf Fibre Reinforced Polypropylene/ Magnesium Hydroxide Composites
Journal of Engineered Fibers and Fabrics

Dear C. H. Lee,

I am pleased to tell you that your work has been accepted for publication in The Journal of Engineered Fibers and Fabrics.

It was accepted on 8 October 2016.

Thank you for submitting your work to our journal. The Jeff Journal Office will contact you with the publication date of your paper.

With kind regards,

Mike Starsinic, Ph.D
Managing Editor
Journal of Engineered Fibers and Fabrics

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CHAPTER 5

MELT VOLUME FLOW RATE AND MELT FLOW RATE OF KENAFFIBRE REINFORCED FLOREON/MAGNESIUM HYDROXIDE BIOCOMPOSITES

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ABSTRACT

A study of the melt volume flow rate (MVR) and the melt flow rate (MFR) of kenaffibre (KF) reinforced Floreon (FLO) and magnesium hydroxide (MH) biocomposites under different temperatures (160-180 ^{o}C) and weight loadings (2.16, 5, 10 kg) is presented in this paper. FLO has the lowest values of MFR and MVR. The increment of the melt flow properties (MVR and MFR) hasbeen found for KF or MH insertion due to the hydrolytic degradation ofthe polylactic acid (PLA) in FLO. Deterioration of the entanglement density at hot temperature, shear thinningand wall slip velocity were the possible causes for the higher melt flow properties. Increasing the KF loadings caused the higher melt flow properties while the higher MH contents created stronger bonding for higher macromolecular chain flow resistance, hence lower melt flow properties were recorded. However, the complicated melt flow behaviour of the KF reinforced FLO/MH biocompositeswasfound in this study. The high probability of KF-KF and KF-MH collisions was expected and there were more collisions for higher fibre and filler loading causing lower melt flow properties.

Keywords: Melt flow rate; Melt volume flow rate; Floreon; Kenaffibre; Magnesium hydroxide; biocomposites

List of Abbreviations

1. Melt volume flow rate (MVR)
2. Melt flow rate (MFR)
3. Kenaf fibres (KF)
4. Floreon (FLO)

5. Magnesium hydroxide (MH)
6. Polylactic acid (PLA)
7. Polyethylene terephthalate (PET)

1. INTRODUCTION

In recent years, natural fibre reinforcement with thermoset or thermoplastic polymer biocomposites have been studied intensively (Sapuan and Harimi, 2003;Sastra, Siregar et al., 2006;Maleque et al., 2007;Aimi et al., 2014). Floreon (FLO) was developed by The University of Sheffield and CPD PLC in November 2013 (blog, 2013). It is a biodegradable polymer which is constructed using standard polylactic acid (PLA). It was created for the greener, safer and better performance of the biopolymer. A lower manufacturing energy is required to produce FLO since it can be processed at about 160^{o}C, while most of the matrices require a temperature higher than 180 ^{o}C (Liang et al., 2011;Ersoy and Taşdemir, 2012;Lee et al., 2014;Shukor et al., 2014;Libolon, 2015). Besides this, it ensures a lower chance of fibre thermal degradation, especially for a low thermal stability natural fibre. FLO is a recyclable and fully biodegradable polymer. Mechanical recycling, as in the case of polyethylene terephthalate (PET), is applicable to FLO. This method requires less energy to reproduce recycled plastic (52.6% less energy for recycling PET) as well as solving the landfill pollution problem (Bioplastics, 2011). On the other hand, feedstock recovery is an alternative option for FLO. This technique is currently applied to PLA and converting its product into the original material (lactic acid). A 99% and above recovery rate has been claimed for PLA (Floreon, 2009). Besides this, in-house testing has shown that FLO has better durability, strength and toughness. In addition, it has four times the impact resistance of PLA cast sheet specimens and almost twice the toughness of PET (Duc et al., 2011).

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Melt flow properties have provided a significant insight for polymer manufacturing. MVR and MFR are indicators of the flow properties of the material in melt. Investigations have been performed with respect to the MVR of PLA composites with different temperatures and loadings (Aimi et al., 2014). A higher MVR value was found with increased temperature due to the increase of the melt free volume. A higher applied load also resulted in a higher MVR due to the shear thinning effect. On the other hand, lower MFR properties of PLA compared to polypropylene (PP) have been found, e.g. poorer wetting on KF, leading to weak fibre-polymer interaction and causing lower strength properties (Han et al., 2012). Also, it has been reported that the viscosity increases with the loading of KF in composites; this is because changes in molecular weight are caused by KF and the interaction between the fibres and the matrix (Mohammad and Arsad, 2013). Another study has been conducted which concerns the effect of MH particle size on the PP matrix (Yang et al., 2009). Decreasing melt flow properties were shown for the composites for particle sizes up to 5 µm, yet an increase in melt flow properties was found for MH particle sizes larger than 5 µm. This is because the small particles enhanced the macromolecular chain flow resistance, while the larger particle sizes reduced the flow resistance as a decreased distance was found between the flame retardant particles (Yang et al., 2009).

From the above reviews, it is evident that no previous work has been conducted on the MFR of the KF reinforced FLO biocomposite with MH used as a flame retardant filler. Therefore, the aim of the present work is to study MH inclusion and the MVR and MFR of KF reinforced FLO biocomposites.

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2. EXPERIMENTAL SECTION

2.1 Materials

The FLO biopolymer Grade 100 was contributed by The University of Sheffield and was used as the matrix. As shown in Figure 61, the differential scanning calorimetry (DSC) curves of previous work indicate the melting peak temperature for the first and second heating cycle. The first heating cycle was intended to remove the thermal history of the polymer. The melting temperature is 150.5 ^{o}C. KF with average length of 8-15 mm was obtained from Tazdiq Engineering, Serdang, Malaysia, in order to reinforce the composites. MH was supplied by Fisher Scientific UK Ltd with 95% purity and was used as a non-toxic flame retardant to enhance the material's fire barrier properties. The sodium hydroxide used in the alkaline treatment was supplied by APC Pure, UK.

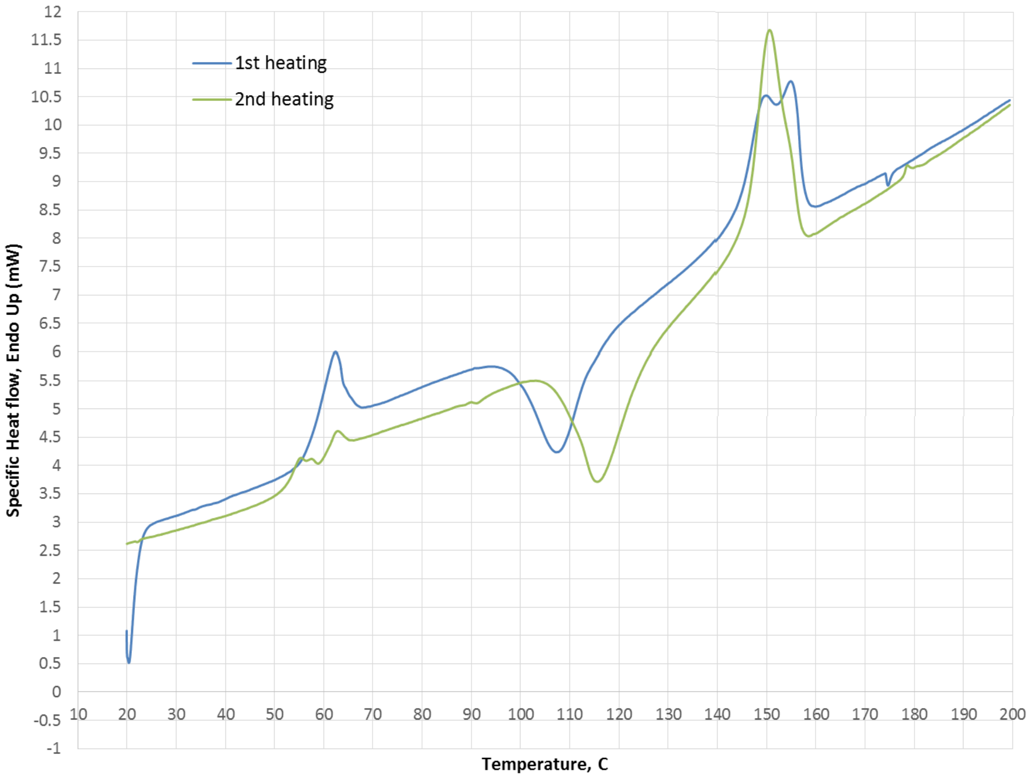

Figure 61. DSC curve of the FLO polymer.

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2.2 Processing methods

Nine samples with different ratios of FLO biocomposite were prepared using a 21 mm lab twin screw extruder (Table ***1534***15). KF was initially dried at 50^{o}C for 24 hours before undergoing 6 % NaOH treatment for 4 hours. Then the KF was washed with water and dried at 100 ^{o}C for 6 hours. All combinations of the composite were simply blended by hand. The extrusion(L/D=30) was performed at 50 RPM and180 ^{o}C at the die head and increasing to 186 ^{o}C at the feed section. The extruded strands were then air-dried and pelletised. The pellets were then tested for their melt flow properties.

Table 1534. Composition of the FLO biocomposites

Sample	Floreon, wt%	Kenaf, fibrewt%	Magnesium Hydroxide, wt%
1	100	-	-
2	95	5	-
3	90	10	-
4	95	-	5
5	90	-	10
6	90	5	5
7	85	5	10
8	85	10	5
9	80	10	10

2.3 Characterisation

2.3.1 Melt flow index testing

The main experimental instrument used in this work was the Mflow extrusion plastometer, which was supplied by Zwick Testing Machines Ltd. The machine was in a laboratory of The University of Sheffield, UK. The melt flow properties of the composites were measured in the temperature range 160 to 180 ^{o}C and for weight loadings of 2.16, 5 and 10 kg. The weight of the die rod was 0.325 kg and the die diameter were 8.26 mm. 300 seconds of pre-heating was conducted after a measured amount of sample was put into the machine’s chamber.

3. RESULTS SECTION

Figure 62 shows the MVR and the MFR of the FLO biocomposites under different loadings at170 ^{o}C. Both the indices (MVR and MFR) increased with the loading applied. Under a high loading, a high shear rate is exerted at the wall along the channel. A common phenomenon known as “shear thinning” exists and this effect is found to be more obvious at higher weight loading. Shear thinning is an effect whereby there is a higher flow rate for increasing shear rate under constant temperature (Liang et al., 2011).

Alkaline treated KF has largely decreased the amount of impurities and hence given a smoother surface to the fibres in the composites. A ball-bearing effect has been performed by the KF and this has resulted in a higher MFR (Liang et al., 1999). When the KF with a random arrangement is applied to the shear, the fibres are forced to align in the flow direction (shear direction). The degree of alignment depends on the shear rates, and a high shear rate causes almost complete alignment and thus higher flow rates (Lafranche et al., 2015). Besides this, a generous portion of the flow velocity was contributed by the wall slip especially for low weight loading. The change in the wall slip velocity is greatly influenced by the fibre content in the composites, rather than merely by the polymer behaviour at the die surface. Previous study showed a 100% wall slip contribution at a low shear level for 60% maple HDPE composites and 40%

pine composites (Li and Wolcott, 2004). More work needs to be done to determine how much flow velocity was contributed by the wall slip theory.

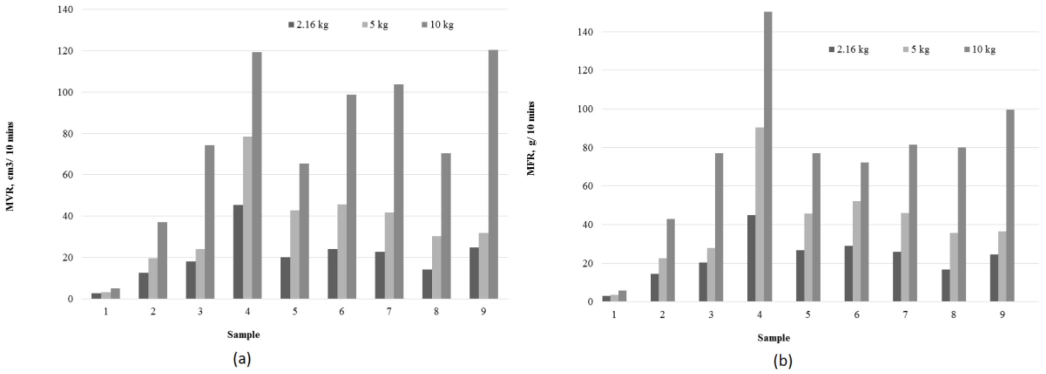

Figure 62. (a) MVR (b) MFR of the FLO biocomposites under different loading at170 °C

Ersoy and Taşdemir (2012) indicated a more than 52.22 % decline of MFR from 0-20 wt% of MH filled composites. By increasing the MH content from 5 to 10 wt% (sample 4 to 5), the MH particles formed new network junctions in the composites, resulting in better interaction forces and friction forces (Crowson et al., 1980;Khalina et al., 2011). Therefore lower MVR and MFR values for the composites were found with increasing MH contents (Liang et al., 2000).

Figure 63 show the MVR and the MFR of the FLO biocomposites under a constant load of 2.16 kg across the temperature range of160 to 180 °C. It can be seen from Figure 63 that both the MVR and the MFR were found to increase with temperature. The polymer molecules absorbed the heat energy and weakened at high temperature. The weakened polymer has a higher free volume in specific weight and hence this resulted in a higher MVR. On the other hand, the higher temperature led to the deterioration of the entanglement density. This caused the sample to flow faster and higher in the MFR since the molecular layers became more slippery (Liang et al., 1999;Lafranche et al., 2015). There was also an increment in the activation energy for the polymer molecules at higher temperature. Hence, an increase in the MFR was found, which agreed with previous work (Gilbert et al., 1982). On the other hand, sample 9 was unable to undergo the test after the pre-heating stage at 180 ^{o}C. The sample expanded significantly in the chamber while pre-heating, restricting it from flowing.

FLO has the lowest flow index values compared to its composites. It is believed that the insertion of the KF and MH disturbed the molecular chain of the FLO. The hydrophilic nature of KF and MH induced the hydrolytic degradation of the PLA in FLO (Li, 1999;Tsuji and Ikarashi, 2004;Baimark and Srihanam, 2015). This reduced the polymer’s molecular length, and flow is easier with a shorter length (Liang and Peng, 2009;Gorrasi and Pantani, 2013). At the same time, a poor interaction between KF and FLO was found in sample 3 using a scanning electron micrograph (Figure 64); they were expected to have a higher flow capability. On the other hand, the insertion of MH (5 wt%) caused a significant increase in the melt flow properties. However, further MH insertion (10 wt%) created new bonding in the composites (Ersoy and Taşdemir, 2012). Therefore, the drop in the values for the melt flow properties indicated that strong bonding has resisted the flow. The complicated melt flow behaviour of KF reinforced FLO/MH biocomposites has been found in this study. The addition of KF to the biocomposites (samples 6 to 7) has been found to increase the MFR at160 ^{o}C but decreased the MFR for temperatures of 170 ^{o}C and 180^{o}C. KF is disoriented in composites and the MH disturbs the converging flow in the die entrance due to the natural fibre reinforced polymer composites. Therefore a high probability of KF-KF/KF-MH collisions is expected and there are more collisions with higher fibre loading, which lowers the MFR (Liang et al., 2010). On the other hand, increasing the MH loading in the MH biocomposites has constantly decreased the MFR and MVR values, showing a stronger bonding in the biocomposites.

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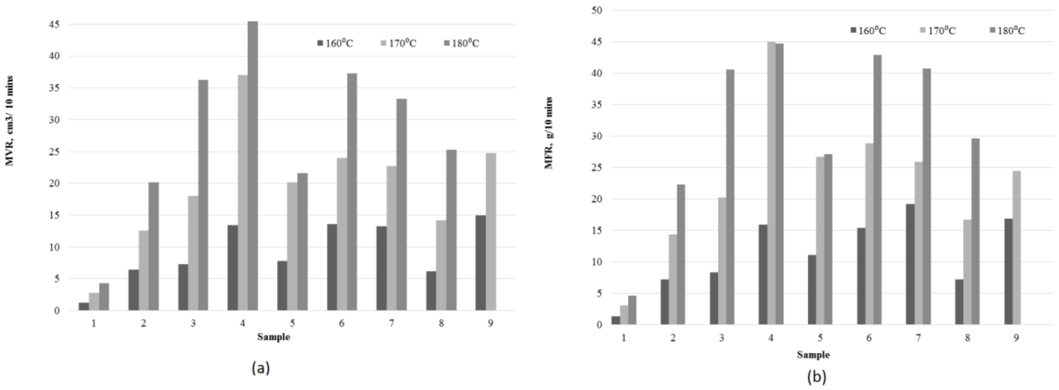

(a)

(b)

Figure 63. (a) MVR (b) MFR of the FLO biocomposites under a constant load of 2.16 kg across the temperature range from 160°C to 180°C.

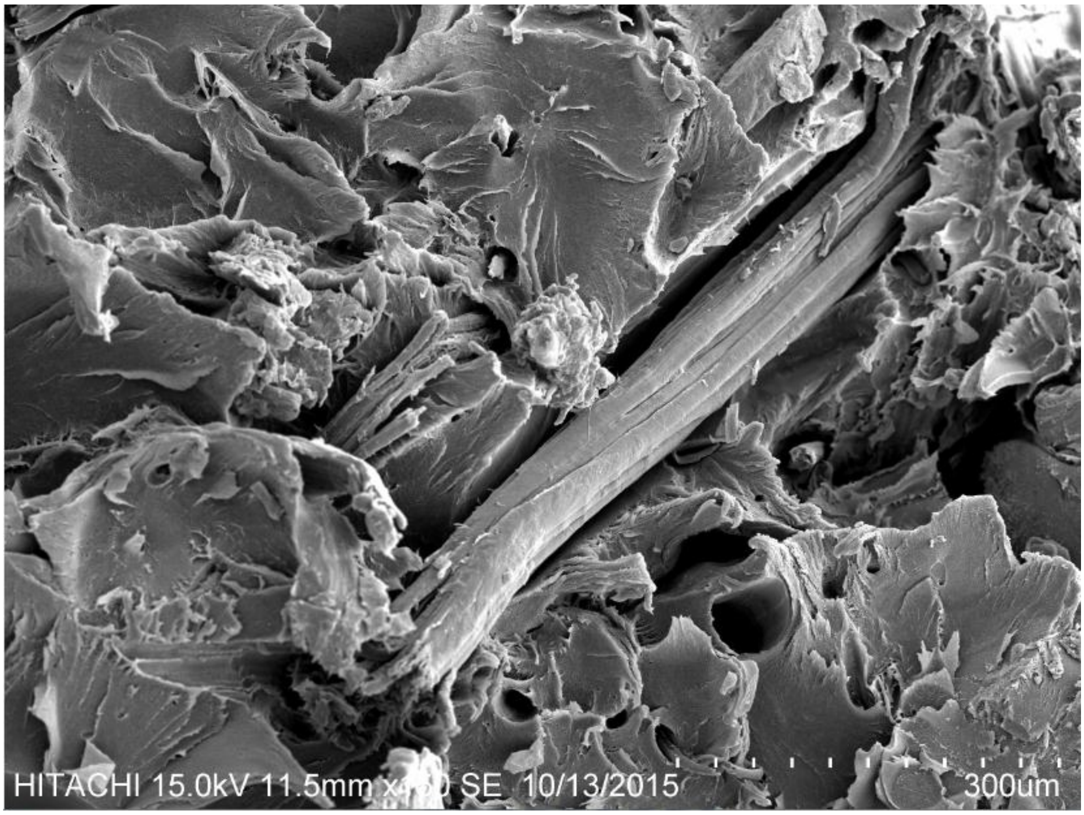

Figure 64. Scanning electron micrograph of sample 3.

4. CONCLUSIONS

The melt flow properties of the KF reinforced FLO/MH bicomposites have been studied for varying temperatures and weight loads. In general, the melt flow capability has been increased for higher temperatures and weight loads. This is because the macromolecular chain has absorbed more heat at higher temperature, causing a weaker

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bonding. On the other hand, the shear thinning effect has been found for higher weight loadings, resulting in higher melt flow properties. FLO has the lowest melt flow indices; it is believed that the hydroxyl groups from the KF and the MH induced hydrolytic degradation on the PLA in FLO. A high content of MH induced new network junctions with better interaction forces and friction forces, causing lower MFR and MVR values. On the other hand, the smooth surface of KF has been forced to align in the flow direction when a load is applied, resulting in higher melt flow properties. Besides this, a large portion of the flow velocity was contributed by the wall slip theory, especially for low weight loading. However, complicated melt flow behavior for KF reinforced FLO/MH biocomposites has been found in this study. A high probability of KF-KF/KF-MH collisions is expected and there are more collisions with higher fibre loading. On the other hand, increasing the MH loading in the MH biocomposites has constantly decreased the MFR and MVR values, showing that the stronger bonding in the biocomposites has resisted the macromolecular chain flow.

AUTHORS’ CONTRIBUTIONS

CHL carried out the experimental work, and the interpretation of the results. CHL, SMS, JHL and MRH discussed and drafted the paper.

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COMPETING INTERESTS

The authors declare that they have no competing interests

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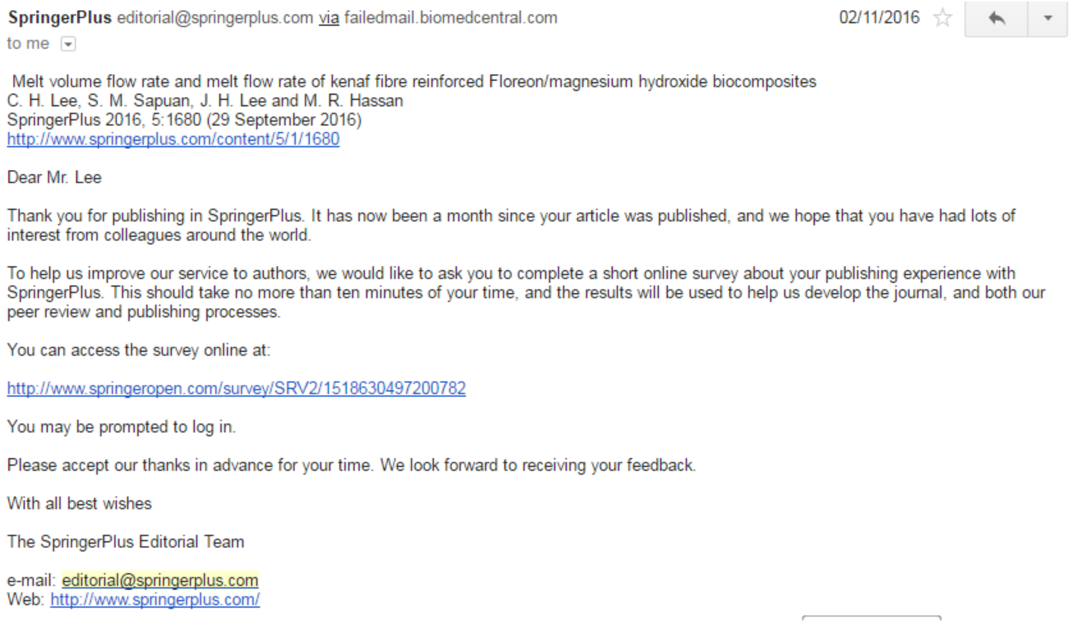

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Melt volume flow rate and melt flow rate of kenaf fibre reinforced Floreon/magnesium hydroxide biocomposites
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Chapter 6

MECHANICAL PROPERTIES OF KENAF FIBRE REINFORCED FLOREON BIOCOMPOSITES WITH MAGNESIUM HYDROXIDE FILLER

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ABSTRACT

This paper presents a study of the mechanical properties ofKenaf fibre (KF) reinforced floreon (FLO)/ magnesium hydroxide (MH) bio-composites. Mixing of all materials by using a 21 mm lab twin screw extruder followed by hot pressing. The composite sheet is then cut into specimens for testing purpose. The Scanning electron microscopy (SEM) was used to study the cross-section of the interface. In this regard, insufficient resin for fibre wetting, hydrolytic degradation on the biopolymer and poor interfacial bonding were attributed to low strength profile. Yet, further addition of KF increased the tensile strength and flexural to 18.91 MPa and 73.09MPa, respectively. Nevertheless, inserting KF and MH filler have found positive outcome onflexural modulus by especially 10KF5MH and 10KF10MH for 3.02GPa and 3.17GPa, respectively. Insertion of KF and MH showed the deterioration of impact strength. However, addition of KF increased the impact strength to 16.82 J/m^2. FLO is a hydrophobic biopolymer, and showed only 0.49% of total water absorption in 14 days. Meanwhile, for the first 24 hours, the rates of water absorption were high very for all bio-composites. Hence, it is worth mentioning that the high contents of KF in bio-composites found higher saturation period and higher total amount of water absorption while the MH causing shorter saturation period but lower total amount of water absorption. However, incompatibility of the interface bonding has increased the water absorption of KF/FLO/MH composites.5KF5MH and 10KF5MH had recorded water absorption of 10.65% and 13.33%. On the other hand, 10KF10MH, were saturated at day 6 with 6.59 % of water absorption. Although 10KF5MH specimen does not have the best performance in mechanical properties, but higher flame retardancy shall provide KF reinforced FLO composite with MH filler for more applications in advance sector especially hazard environment.

Keywords: Kenaffibre;Floreon;Biocomposites; Mechanical properties; Magnesium hydroxide

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INTRODUCTION

Floreon (FLO) is an advanced novel biopolymer, which wasrecently invented in November 2013 (blog). The FLO is mainly composedof standard polylactic acid (PLA), hence, it is equipped with biodegradable properties. Furthermore, 160 °C of processing temperature provides a lower production energy andlow chancefor fibre thermal degradation(Tang and Liang, 2003;Liang et al., 2011;ERSOY and TAŞDEMİR, 2012;Shukor et al., 2014). These features have garnered much interest from the public.

Other than being fully biodegradable and easily decomposed into soil, the FLO can undergo mechanical recycling by using lower energy. Itis to reproduces biopolymer that can reduce land waste problem. In this regard, only half of production energy is needed(Libolon, 2015). The FLO had also been proved to have better toughness, strength and durability, as compared to conventional polymer (Floreon, 2009). In the meantime, biopolymer could loss the strength when ultraviolet (UV) degradation. FLO has a good UV stability and hence been promoted for the use in lithographic printing.

In this light, natural fibres are commonly selected as composite reinforcement to enhance performance (Sapuan et al., 2003;Sastra et al., 2006;Maleque et al., 2007;Zainudin et al., 2009;Davoodi et al., 2011;Ibrahim et al., 2012;Ismail and Aziz, 2015;Fairuz et al., 2016;Mohammed et al., 2016;Salleh et al., 2013).Solving for environmental issues and better tool life were the main reasons being selected(Duc et al., 2011;Al-Oqla et al., 2015). Besides, it is expected to produce composite with lower density and cheaper cost (El-Shekeil et al., 2014). However, unfortunately, many factors have caused fibres to differ from each other. Inherently, inconsistencies of the component composition are the main reasons for wide range of properties(Charlet et al., 2010).

A previous study found that kenaf fibre (KF) reinforced PLA biocomposites have better tensile properties compared to pure PLA matrix (Ochi, 2008). This showed that the KF has a good interfacial bonding with PLA matrix. Another studyconducted some experiments towards KF reinforced PLA biocomposites on basic mechanical properties (Flexural properties, impact resistance and compression properties) as a potential material for construction material(Srebrenkoska et al., 2006). The results obtained indicated that the properties of KF reinforced PLA biocomposites are superior to most types of traditional building composites. However, higher water absorption behaviour was expected due to the hydrophilic nature KF. Apart from that, voids presented between KF and PLA had increased the water uptake behaviour. These voids were caused by the poor interfacial bonding between the KF and PLA (Anuar et al., 2010). To reduce these voids, alkalization treatment by using sodium hydroxide on KF has improved the mechanical properties compared to untreated short KF. The 6% concentration of sodium hydroxide was found optimum in terms of cleaning the fibres surfaces (Mwaikambo and Ansell, 2002;Meon et al., 2012).

On the other hand, insertion of flame retardant filler into KF reinforced PLA biocomposites had deteriorated the tensile strength (Cho et al., 2014). It is believed that the flame retardant has caused microstructural defects in the matrix. Besides that, a degradation of polymer is always accompanied by a reduction of its molecular weight while magnesium hydroxide (MH) was reported to be a cursor on controlling the polymer degradation rate (Mobedi et al., 2006), where the insertion of MH into PLA matrix had increased the rate of degradation due to the lower molecular weight found in MH contented composites. Thus, it was concluded that the higher MH concentration in PLA matrix had caused a higher rate of PLA degradation. In addition, lower strength properties were expected for low molecular weight polymer (Mobedi et al., 2006).

From the reviews above, it is evident that there is no previous work conducted on the mechanical properties of KF reinforced FLO composite, particularly by using MH as flame retardant filler. Therefore, the aim of the present work is to study the effects of MH inclusion on the mechanical properties of KF reinforced FLO composites.

EXPERIMENTAL SET UP

Materials

The polymer used in this work was Floreon (FLO) resin Grade 100, which was supplied by The University of Sheffield. The short kenaf fibre (KF), with the average length of 8-15mmwas used as the reinforcement fibre and was supplied by Tazdiq Engineering (002203302-V). Meanwhile, non-toxic flame retardant to enhance the fire barrier properties came from the95% purity ofAcros Organics Magnesium hydroxide (MH) which was purchasedfrom Fisher Scientific UK Ltd while the sodium hydroxide (NaOH) used in the alkali treatment was contributed by APC Pure, UK.

Processing of composites

Table ***1635***16 lists the different ratios of FLO composite samples, which were prepared using a 21 mm lab twin screw extruder. Here, KFs were dried for 24 hours at 50 ^{o}C before treated with 6 % NaOH, for 4 hours. Then, it was washed and rinsedby using water and dried for 6 hours at 100^{o}C. The working temperature for the extrusion during compounding was set at 180 ^{o}C at the die head and was increased to 186 ^{o}C at the feed section with 50RPM. The process method was adapted from a previous study (Zhang et al., 2012). The extruded strands were then dried by blowing air before being pelletised. The pellets were then carried to a hot press machine in The University of Sheffield Csic laboratory for sheet fabrication. The setting of the hot press machine was set to five bar of pressing pressure for 10 minutes, before going through 10 minutes of pre-heating. The mould was then allowed to cool for 10 minutes in room temperature. The process flow is shown in Figure 65.

Table 1635. Combination formula of KF/FLO/MH composites.

Sample name	Floreon (FLO), wt%	Kenaffibre (KF), wt%	Magnesium Hydroxide (MH), wt%
Pure FLO	100	-	-
5KF	95	5	-
10KF	90	10	-
5MH	95	-	5
10MH	90	-	10
5KF5MH	90	5	5
5KF10MH	85	5	10
10KF5MH	85	10	5
10KF10MH	80	10	10

Characterisation Techniques

Scanning electron microscopy (SEM) was used to study the morphology of the specimen where the cross-sections of flexural testing specimen represent the location of study. The accelerating voltage of 15kV helped to image the cross-sections.

Tensile tests were carried out with ASTM D638-14 using the Instron 5kN machine locate in Universiti Putra Malaysia (UPM) (ASTM D638-14, 2014). A One mm thick dumbbell specimen was cut from the moulded sheet while a cross-head speed of

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1mm/min was used and the test was performed at 25 °C. Flexural testing was conducted using the same Instron 5kN machine in UPM according to ASTM D790-10 testing standard (Venkateshwaran and Elayaperumal, 2010). The dimension of the specimen was 96 mm × 12.7 mm × 5mm, which included 10% of the support span for overhanging.

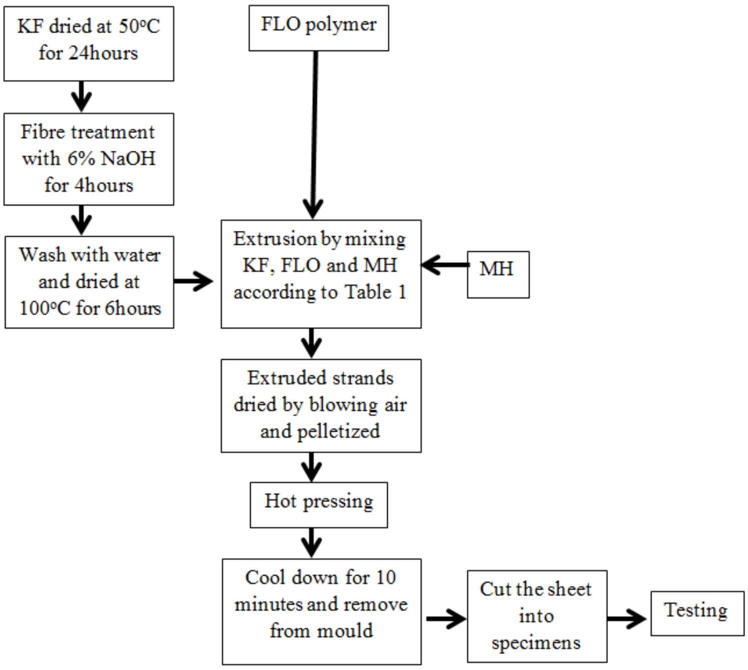

Figure 65. Process flow chart.

The un-notched specimens were subjected to Charpy impact testing which adheres to ASTM D6110-10 (2010). The dimension of the test specimen was 127 mm × 12.7 mm × 5 mm. Each specimen was measured with Vernier Caliper before the impact testing was conducted where the accuracy of the reading was up to 0.1 mm. The specimen was then positioned horizontally on the supports of the impact testing machine. The pendulum was raised and secured in the release mechanism and the indicating board was reset. The pendulum was then released to provide impacts to the specimen; consequently, the indicated readings were recorded for impact strength calculation.

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Meanwhile, to test water absorption, the test specimen was initially dried at 50 ^{o}C for 24 hours by following ASTM D570-98(2010)e1, (2010). The specimens were then placed so that they were entirely immersed in a container of distilled water which was prepared by the UPM chemical laboratory. At the end of the first, second and 24th hours, the specimens were removed from the water and all their surfaces were wiped off with a dry cloth. Here, the weighing scale was nearest to 0.001g and one specimen was put back into the container before the next one was taken out and the process was continued until all the specimens had been processed. After that, these drying and weighing procedures were repeated for every 24 hours until day 7. Then, the repeating duration was changed to every weekly (7 days) until all specimens had been saturated. In this light, each specimen was considered as saturated when only less than 1 % of its total weight (5mg) increased. In all, five specimens were tested for each group, and average results were recorded and reported.

RESULTS AND DISCUSSION

SEM morphology

The scanning electron micrographs for all samples, except the pure FLO polymer, are shown in Figure 66. The chemical treatment of the fibre surface had created good interfacial bonding between the fibre and the matrix (Figure 30 a, b). Meanwhile, Figure 30(c) and Figure 30(d)show a coarser surface under SEM due to the insertion of the MH fillers, which is similar to what found in a previous study a result of severe agglomerations and less uniform distribution of MH filler (Aziz et al., 2012). On the other hand, Figure 30e-h show the formation of voids between the fibre and the matrix. This high degree of pull-out indicates poor interfacial bonding(Anuar et al., 2011). In this light, the deterioration of mechanical properties was expected(Suharty et al., 2012;Lee et al., 2014).

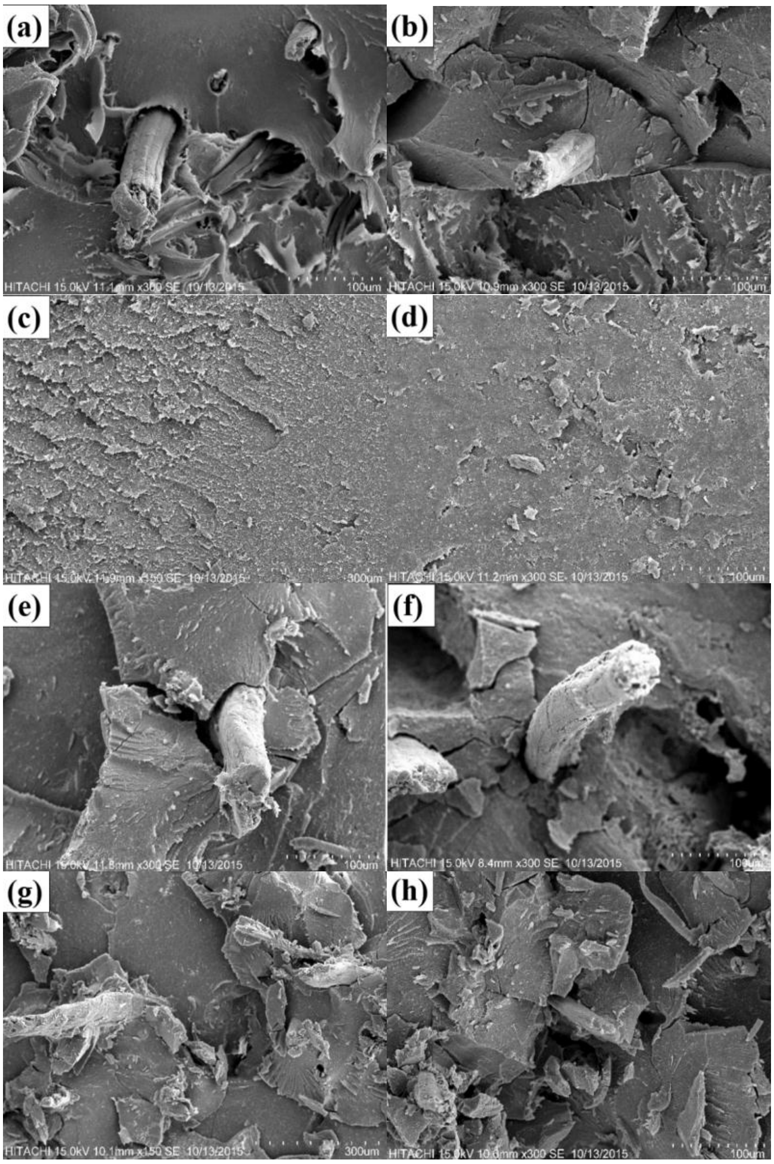

Figure 66. SEM micrographs of the (a) 5KF; (b) 10KF; (c) 5MH; (d) 10MH; (e) 5KF5MH; (f) 5KF10MH; (g) 10KF5MH; (h) 10KF10MH.

Tensile Properties of composites

Figure 67 and Figure 68 show tensile strength and tensile modulus for KF reinforced FLO composites with MH fillers. The net FLO polymer presents the best tensile strength compared to other samples compared to other specimens. This is due to poor interfacial adhesion between fibre and matrix and the voids created (Lee et al., 2009).

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The low fibre content has shown a lower load transfer ability, and then the stress was accumulated within matrix, causing the composite to fail(Salman et al., 2016).Therefore insertion of 5wt% of KF did not improve the tensile strength. However, the tensile strength had increased with the increase of fibre loading. The tensile strength had increased by 25.2 % from 15.1 MPa to 18.9 MPa for 5KF to 10KF as there are more reinforcement fibre content in 10KF, to withstand more load before breaking compared to 5KF (Ishak et al., 2010). This suggests that the fibre dispersion and distribution had started to improve. Similar results indicated the tensile strength shown significant improvement for fibre volume more than 30 wt% (Tawakkal et al., 2014). Hence, Ibrahim, (2010) concluded that 30 wt% of KF is the optimum loading for PLA biopolymer. Furthermore, the insertion of the MH showed a negative impact on the tensile properties,where the MH has found to be a cursor for controlling the polymer degradation rate, and accelerates the rate of degradation of composite (Mobedi et al., 2006). The highest drop (56.5 %) in tensile strength was found for 10KF10MH, but not in10KF5MH. Besides that, high fibre and filler contents in the composites had caused insufficient resin to transfer the load applied (Datta and Kopczyńska, 2015). A bad wetting affects the load transfer mechanism in the composite, leading to low strength properties. Insufficient resin also found to face difficulties to maintain its structure. In this case, the sample would fail before it could reach the highest absorbable strength of the fibre. On the contrary, the highest modulus was achieved by 10KFas agreed by previous studies (Lee et al., 2009). On the other hand, MH insertion reduced the modulus of composites, regardless of KF contents in the composites. Other factors like porosity of composites and composite's crystallinity were also accounted to the variations of the stiffness and strength of the composite (Nor Azowa Ibrahim et al., 2010).

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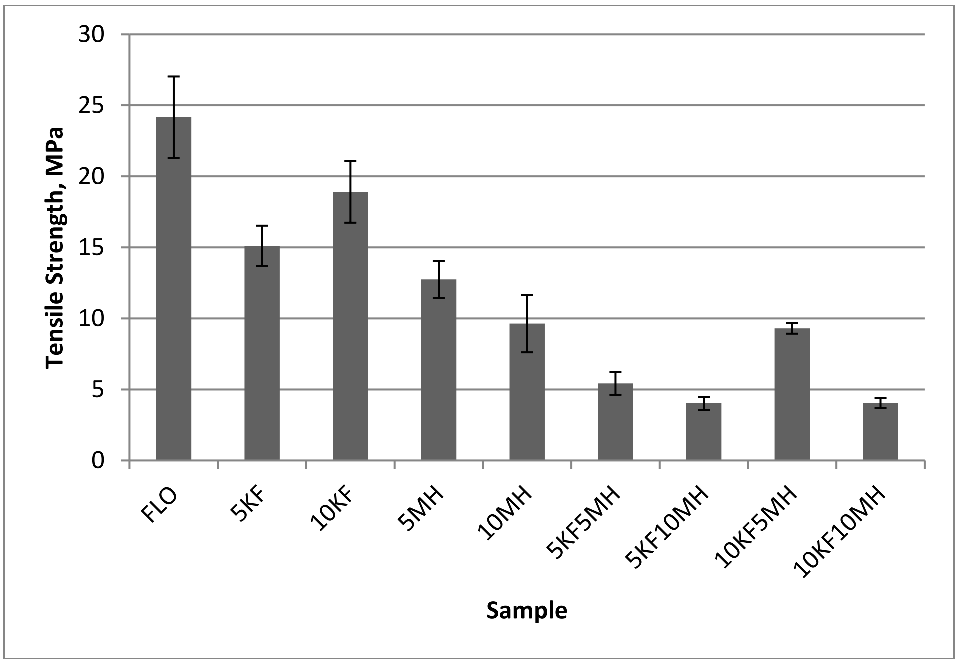

Figure 67. Tensile strength for the KF reinforced FLO composites with MH fillers.

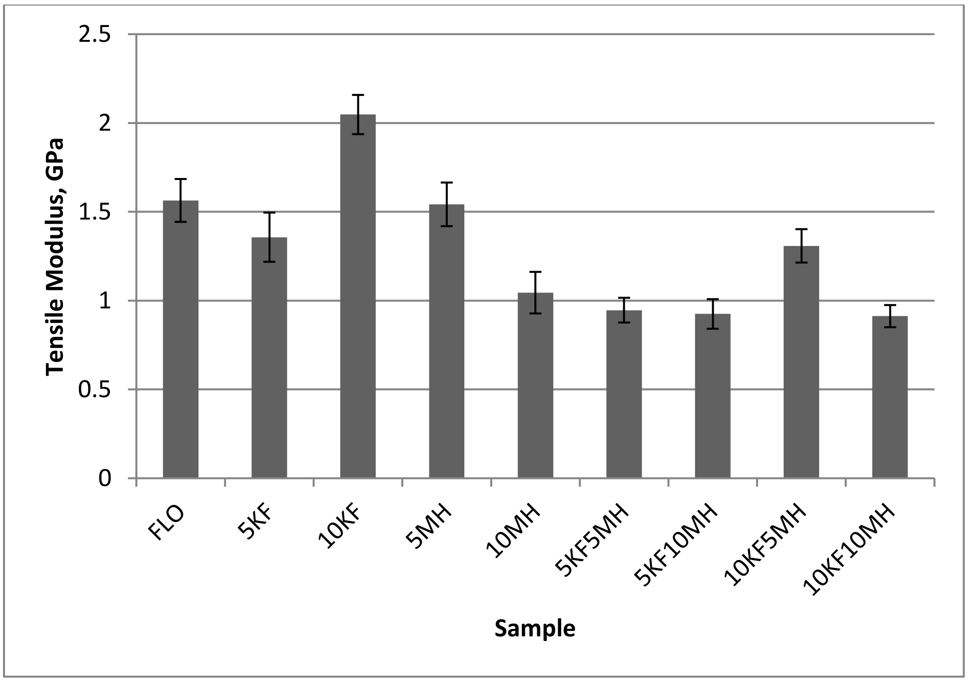

Figure 68. Tensile modulus for the KF reinforced FLO composites with MH fillers.

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Flexural Properties of composites

Figure 69 and Figure 70 show the flexural strength and flexural modulus for the KF reinforced FLO composites with MH fillers, respectively. The flexural strength of the FLO is the highest amongst all composites and the reduction of flexural strength was recorded after the insertion of KF and MH filler. Hydrolytic degradation, which is known as random hydrolysis on the PLA, was agreed by previous studyas a reason for lower flexural strength (Tsuji and Ikarashi, 2004). Meanwhile, the shorter molecular chain of the FLO had decreased its ability to withstand the bending force (Gorrasi and Pantani, 2013;Baimark and Srihanam, 2015). Asthe higher loading of the hydrophilic KF contains more hydroxyl groups to induce hydrolytic degradation, hence, further deterioration of the flexural strength was found. The flexural strength had reducedby 29.1%, from pure FLO to 5KF. However, it had increased by 24.4% from 5KF to 10KF. The higher strength properties were contributed by addition of KF. However, High temperature of the thermo-processing had induced some chain scissions to occur in the biopolymer. On the contrary, the degradation becomes fasteras the MH concentrations increased(Mobedi et al., 2006). Meanwhile, the flexural strength has reduced by 32.6 % and 54.1% for pure FLO to 5MH and to 10MH, respectively. Besides that, the low compatibility of the KF and MH filler on the FLO was another factor for thereduction in flexural strength. This is because the load transfer within the composite was not working well under the low compatibility condition. A similar finding was reported by other study (Sain et al., 2004). On the other hand, the flexural modulus of the composites was found to be affected by the KF and MH, wherethe insertion of KF and MH filler will gradually increase its flexural modulus. The 10KF5MH and 10KF10MH showed a positive result on flexural modulus where both have higher flexural modulus values of more than 3.00GPa, indicating the rigidness.

The differences between values of tensile modulus and flexural modulus have found identical in past study, showing difference trending of tensile and flexural modulus (Lee et al., 2009). It was found that different in the heterogeneous cross section and shear deformation between the tensile and flexural test, have created differences in modulus value (Tolf and Clarin, 1984).

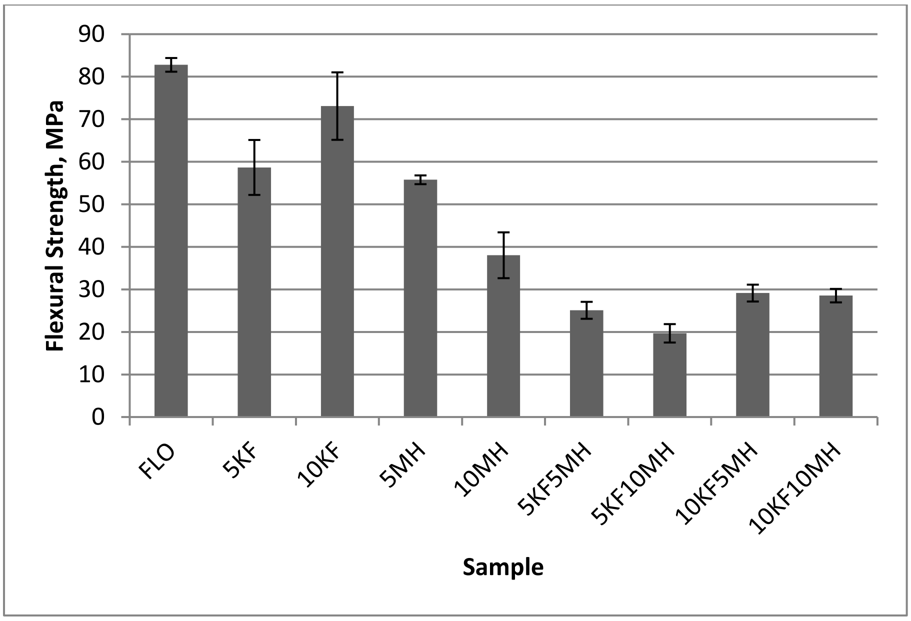

Figure 69. Flexural strength of the KF reinforced FLO composites with MH fillers.

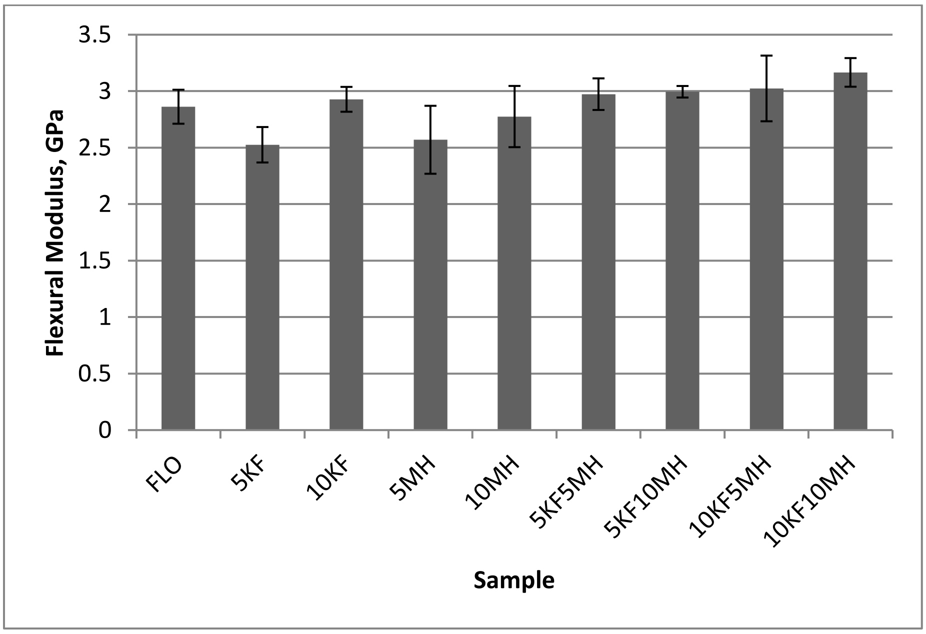

Figure 70. Flexural modulus of the KF reinforced FLO composites with MH fillers.

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Impact Properties of composites

Impact strength is a measurement of the capability of a material to absorb the energy, which occurs when a sudden load was applied to the materials. Thus, un-notched Charpy impact testing was conducted and the results are shown in Figure 71. During the testing, all of composite samples were broken completely into two parts due to the brittle nature of the biopolymer (Bajpai et al., 2012). In this light, the biopolymer is a significant factor for analysing the impact strength. Brittle biopolymer has exhibited low impact strength (Manshor et al., 2014).However, high impact strength of the FLO was expected and highlighted by the manufacturer(Floreon, 2009).

On the other hand, the reinforcement fibre absorbs the energy for debonding and pull-out, which helped to enhance the impact strength of the composite. Therefore, a good interfacial bond between the fibre and the matrix is important (Alavudeen et al., 2015). Furthermore, although a low content of fibre in the reinforced composite has lower impact strength than pure FLO, yet, the increment of the KF loading in the composites (5KF to 10KF) had increased the impact strength by 49.62%,which has also been noted in previous work (Awal et al., 2015). As this study used short fibres as the reinforcement, consequently, the high amount of fibre end acts as a stress concentration point, creating uneven energy transfer throughout the composite (Yang et al., 2004). Besides that, the random distribution of the short KF, as can be seen from Figure 66, had caused lower impact energy values. On the contrary, the impact strength has reduced 5.8 %, 31.8 % and 11.4 % for 5MH to 10MH, 5KF5MH to 5KF10MH and 10KF5MH to 10KF10MH, respectively. Such reduction of the impact strength was expected with the insertion of MH to the KF reinforced FLO composites (Jeencham et al., 2014). The weak bonding between KF-MH, KF-FLO and MH-FLO has reduced its impact strength.

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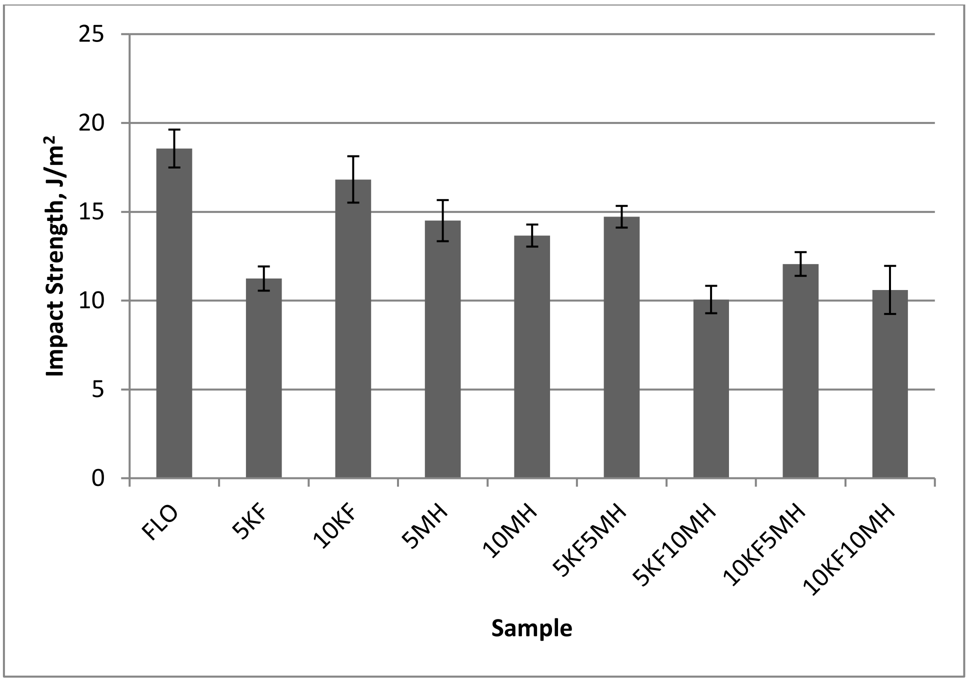

Figure 71. Impact strength of the KF reinforced FLO composites with MH fillers.

Water absorption behaviour of composites

The effects of the KF content and the MH loading on the water absorption in 24 hours, 14 days and until saturation are shown in Table ***17361***7. The rate of water absorption was very high for the first 24 hours and until it reached saturation. The water absorption of all composite samples was higher compared to the water absorption of FLO. This is because FLO is a hydrophobic biopolymer, which showed only 0.49% of total water absorption in14 days. Inherently, the insertion of the KF and MH had shifted the composite to more hydrophilic in nature.

Moreover, in the first 24hours, none of the samples had reached the saturated water uptake. However, 5KF10MH, 10KF5MH and 10KF10MH had recorded 5.78 %, 3.52 % and 2.95 % of water absorption respectively. This high absorption rate was due to the high void content in the samples which was caused by the incompatibility of the interface bonding. Several samples, including FLO had reached saturation after 24

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hours. Furthermore, 5KF10MH and 10KF10MH, were saturated at day 6 with 9.83 % and 6.59 % of water absorption respectively. In the meantime, 10MH and 10KF5MH were found saturated at day 21 with a significant difference in total water absorption. These differences were mainly due to the insertion of hydrophilic KF in the 10KF5MH, which induced an extra 11.7 % of water absorption before saturation. The highly cellulose content of KF contents hydroxide group, which is hydrophilic in nature, had also caused in high water absorption of the specimen(Tan et al., 2014). Annuar, 2010 also confirmed that the voids presented between KF and PLA had increased the water uptakes (Anuar, Ahmad et al., 2010). Consequently, 5KF and 10KF, which constitute of KF reinforced biocomposites, had shown steady water absorption up to day 63. The composite with higher KF content took a longer time to reach saturation but had higher total water absorption, as confirmed by Rashdi (2009).

It is also worth mentioning that the high content of MH fillers contributes to shorter saturation time and lower total water absorption. In this regard, 5MH was saturated at day 35 with 5.94 % water absorption while 10MH was saturated at day 21 with only 1.63 % of water absorption. This may be due to the hydroxyl group in the MH formulation which had strongly attracted water and caused faster rate of water absorption. However, the fine powder of the MH had attributed to the lower void content and lesser empty space for water absorption.

CONCLUSIONS

The scanning electron micrographs reviewed that the agglomeration of MH fillers and formation of voids were due to poor interfacial bonding on the cross-section of specimens. Meanwhile, insufficient resin for fibre wetting, polymer hydrolytic degradation and low compatibility between KF, MH and FLO were attributed to the

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low tensile properties and flexural strength. However, the tensile strength had increased with the increased fibre loading due to better fibre dispersion, while the insertion of KF and MH especially 10KF5MH and 10KF10MH, have shown positive observation on flexural modulus indicating good modulus on bending. In this light, a past study found similar differences between values of tensile modulus and flexural modulus which indicates difference in the trending of tensile and flexural modulus. It was concluded that difference in the heterogeneous cross section and shear deformation between tensile and flexural test, created differences in modulus value. On the other hand, the increased content of KF in composites caused an increment of impact strength, while the insertion of MH flame retardant fillers insertion caused a decreasing trend for impact strength and the impact strengths were reduced by 5.8 %, 31.8 % and 11.4 % for 5MH to 10MH, 5KF5MH to 5KF10MH and 10KF5MH to 10KF10MH, respectively. Besides that, the rates of water absorption for all samples were very high in the beginning but all had reached saturation by different rate. It is worth mentioning that the high content of the KF produced a higher total water absorption but slower saturated composite while the insertion of MH caused faster saturation and lower total water absorption. However, incompatibility of the interface bonding increased the water absorption of KF/FLO/MH composites and even though 10KF5MH specimen does not have the best performance in mechanical properties, the higher flame retardancy shall provide KF reinforced FLO composite with MH filler for more applications in the advanced sector, especially in hazardous environment.

RECOMMENDATIONs AND FUTURE DEVELOPMENTs

It was found that insertion of KF and MH filler had caused significant changes in composite properties. Therefore, it is important to take extra care in every process, especially specimen fabrication. In future development, different types of fibre pre-treatment and fillers could be applied on FLO polymer to investigateits properties for more advanced application. In this light, nano-sized KF and MH fillers are highly recommended to be applied in FLO polymer as they can enhance the composite properties with lesser content volumes, compared to conventional KF and MH.

ACKNOWLEDGEMENT

The authors wish to thank the Universiti Putra Malaysia (UPM) and The University of Sheffield for their support in carrying out this experiment regarding sample fabrication. The authors also wish to express their appreciation to Mr. Fairuz for supplying the materials, and Mr. Wildan and Mr. Ismail for supporting the research.

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Table 1736. Results of water absorption.

Specimen	Water absorption, %								
	1h	2h	24h (Day1)	48h (Day2)	72h (Day3)	96h (Day4)	120h (Day5)	144h (Day6)	168h (Day7)
FLO	0.01	0.03	0.21	0.32	0.34	0.36	0.39	0.40	0.49
5KF	0.07	0.14	0.59	1.02	1.33	1.49	1.82	1.98	2.20
10KF	0.21	0.27	0.81	1.30	1.68	1.94	2.45	2.71	2.93
5MH	0.10	0.17	0.55	1.06	1.40	1.65	2.14	2.38	2.73
10MH	0.10	0.12	0.31	0.52	0.71	0.82	1.06	1.17	1.35
5KF5MH	0.07	0.20	1.18	2.02	2.67	3.05	3.82	4.21	4.69
5KF10MH	1.30	1.71	5.78	7.58	7.71	8.24	9.30	9.83	-
10KF5MH	0.49	0.66	3.52	5.67	7.07	7.77	9.16	9.86	10.41
10KF10MH	0.41	0.55	2.95	4.73	5.83	6.02	6.40	6.59	-

Table 17. Results of water absorption*(cont.)*.

Specimen	Water absorption, %							
	336h (Day14)	504h (Day21)	672h (Day28)	840h (Day35)	1008 (Day42)	1176h (Day49)	1344 (Day56)	1512h (Day63)
FLO	-	-	-	-	-	-	-	-
5KF	2.79	3.22	3.56	3.90	4.08	4.23	4.45	4.57
10KF	3.80	4.29	4.63	4.98	5.13	5.25	5.47	5.68
5MH	4.16	5.40	5.67	5.94	-	-	-	-
10MH	1.56	1.63	-	-	-	-	-	-
5KF5MH	6.68	8.31	9.28	10.25	10.49	10.65	-	-
5KF10MH	-	-	-	-	-	-	-	-
10KF5MH	12.46	13.33	-	-	-	-	-	-
10KF10MH	-	-	-	-	-	-	-	-

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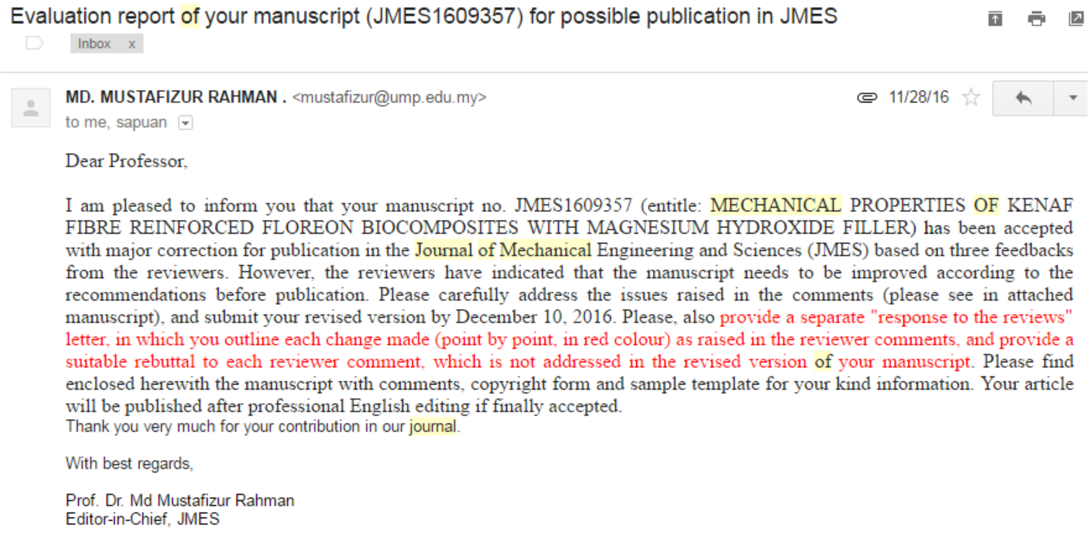
Acceptance email



Evaluation report of your manuscript (JMES1609357) for possible publication in JMES

MD. MUSTAFIZUR RAHMAN . <mustafizur@ump.edu.my>
to me, sapuan

Dear Professor,

I am pleased to inform you that your manuscript no. JMES1609357 (entitle: MECHANICAL PROPERTIES OF KENAF FIBRE REINFORCED FLOREON BIOCOMPOSITES WITH MAGNESIUM HYDROXIDE FILLER) has been accepted with major correction for publication in the Journal of Mechanical Engineering and Sciences (JMES) based on three feedbacks from the reviewers. However, the reviewers have indicated that the manuscript needs to be improved according to the recommendations before publication. Please carefully address the issues raised in the comments (please see in attached manuscript), and submit your revised version by December 10, 2016. Please, also provide a separate "response to the reviews" letter, in which you outline each change made (point by point, in red colour) as raised in the reviewer comments, and provide a suitable rebuttal to each reviewer comment, which is not addressed in the revised version of your manuscript. Please find enclosed herewith the manuscript with comments, copyright form and sample template for your kind information. Your article will be published after professional English editing if finally accepted.
Thank you very much for your contribution in our journal.

With best regards,

Prof. Dr. Md Mustafizur Rahman
Editor-in-Chief, JMES

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Chapter 7

6. THERMAL ANALYSIS OF KENAF FIBRE REINFORCED FLOREONBIOCOMPOSITES WITH MAGNESIUM HYDROXIDE FLAME RETARDANT FILLER

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ABSTRACT

The Floreon (FLO) biopolymer is an advanced bioplastic material, invented by The University of Sheffield and CPD Plc, in November 2013. Nine combinations of the kenaf fibre (KF) reinforced FLO with magnesium hydroxide (MH) flame retardant filler were fabricated and tested on Thermogravimetry Analysis (TGA), Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA). Scanning electron microscopy (SEM) has been used to study the cross-section of interface. The low thermal stability of natural fibre composite has found lower decomposition temperature but a higher residual mass. MH filler containing's composite has higher residual mass at 600 ^{o}C but it is not the best flame retardant for the FLO biopolymer composites as the pure FLO biopolymer has higher decomposition temperature than MH reaction temperature. Some synergistic effect located in char formation, T_g reduction and a lower tan δ peak shown in the three-phase system (KF/FLO/MH). The MH filler has found more significant in enhancing mass residual. The T_g were show deterioration for all samples compared to the pure FLO biopolymer. The melting temperature has found no momentous change either KF or MH or both of these were inserted. The values of co-coefficient, C recorded decreasing as increasing the fibre loading. This showing the fibres transfer the loading effectively. Close value of storage moduli found in DMA for all samples except sample 4.

Keywords: Kenaf fibre, Floreon Biopolymer, Thermogravimetry Analysis, Differential Scanning Calorimetry, Dynamic Mechanical Analysis

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INTRODUCTIONThe University of Sheffield and CPD plc invented Floreon (FLO), an advanced bioplastic material, in November 2013 (Blog, 2013). Polylactic acid (PLA) was the key ingredient in the FLO biopolymer and therefore its biodegradable behaviour is promising. The low thermal stability of natural fibre easily causes fibre thermal degradation in thermal processing. The low processing temperature (160^{o}C) of the FLO biopolymer could prevent this issue and most importantly lower the production energy (Tang and Liang, 2003;Liang et al., 2011;Ersoy and Taşdemir, 2012;Shukor et al., 2014).

A mechanical recycling method could be applied with FLO, rather than being fully biodegradable and decomposed into the soil. This alternative is also available for polyethylene terephthalate (PET). It reproduces recycled biopolymer using lower energy, as well as lessening landfill pollution. The production of recycled PET has been found to achieve a 52.6% energy saving by mechanical recycling (Libolon, 2015). Besides this, the feedstock recovery method is an alternative way to recycle the product without losing its properties. This method was previously used on PLA, converting it into lactic acid and producing virgin PLA again. Research has claimed that more than 99% of the recovery rate has been achieved for PLA (Bioplastics, 2011).

FLO has been shown to have better strength and durability compared to standard PLA. Besides this, having twice the toughness value and four times the impact resistance properties of PET and PLA have widened the use of FLO as synthetic biopolymer substitution (Floreon, 2009). Ultraviolet (UV) degradation causes the biopolymer to loss its properties. FLO successfully enhances its UV stability and is introduced in lithographic printing which is exposed to UV light.

To perform in advanced sectors, outstanding properties are required on top of biodegradable behaviour. Natural fibres have famously been selected as a composite reinforcement to improve its performance (Sapuan and Harimi, 2003;Sastra et al., 2006;Maleque et al., 2007;Zainudin et al., 2009;Davoodi et al., 2011). Natural fibres have been selected as they produced lower environmental issues and prolong the period of tools (Duc et al., 2011;Al-Oqla et al., 2015). At the same time, they have extraordinary properties with lower cost and density (El-Shekeil et al., 2014). Unfortunately, different growth conditions, processing and extraction methods caused every single fibre to differ from each other. The difference in natural fibre properties is mainly because of the inconsistency of the component composition (cellulose, hemicellulose, lignin, pectins, wax and ash) (Charlet et al., 2010).

Among many types of natural fibres, kenaf (Hibiscus cannabinus L.) fibre is one of the well-known natural fibres under intensive research as a reinforcement for composites. KF could be produced two or three times annually in a wide range of conditions (Saba et al., 2015). The kenaf plant is able to absorb the phosphorus and nitrogen minerals in the soil and this helps to increase the growth rate (Kuchinda et al., 2001). In addition, the almost threefold photosynthesis rate of the kenaf plant over conventional trees has helped in oxygen production (Nishino et al., 2003).

Natural fibre could be either reinforced in thermoplastic or thermoset. Both have brought different performances and hence have used in different applications. High moisture absorption, fibre aggregates tendency and incompatibility between natural fibres and the matrix have become the significant factors for advanced materials (El-Shekeil et al., 2011). Surface modification has been made for better interfacial bonding between natural fibres and the matrix. A good interfacial bond prompts better load transfer and thus better properties (Ku et al., 2011;Reid et al., 2011;Asumani et al., 2012;Ren et al., 2012;Yousif et al., 2012;Zheng et al., 2013;Osman et al., 2014;Yousfi et al., 2014;Yahaya et al., 2015).

The aim of the present work was to study the thermal and morphological properties resulting from the kenaf fibres (KF) and magnesium hydroxide (MH) used in the FLO biopolymer. The FLO biopolymer was chosen because of its biodegradability and favourable properties. MH is a non-toxic flame retardant filler, which has a highly potential to increase the fire barrier properties of composites. KF is one of the natural fibres with a fast-growing, high photosynthesis rate and high strength properties. The alkaline treatment is proposed for KF. This treatment was studied previously, and the results were undeniably impressive. It reduces the impure substances and produce more active sites in every single fibres for higher properties by better adhesion between the fibres and matrix.

MATERIALS AND METHOD

Materials

The Floreon (FLO) resin Grade 100 used in this work was supplied by The University of Sheffield. The reinforcement fibre used was kenaf fibre (KF) and this was bought from Tazdiq Engineering (002203302-V). The Acros Organics magnesium hydroxide (MH) was supplied by Fisher Scientific UK Ltd with 95% purity. It was used as a non-toxic flame retardant to enhance the fire barrier properties. The sodium hydroxide (NaOH) used in the alkaline treatment was supplied by APC Pure, UK.

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Processing of composite materials

Samples of different Floreon (FLO)/Kenaf Fibre (KF)/Magnesium Hydroxide (MH) ratios were prepared using a 21 mm lab twin screw extruder and listed in Table ***1837***18. KF were initially dried at 50^{o}C for 24 hours before treatment with 6 % sodium hydroxide (NaOH) for 4 hours. Then the fibres were washed with water and dried 100^{o}C for 6 hours. The extrusion was performed at 50 RPM. The working temperature during compounding were 180^{o}C at the die head and increased to 186^{o}C at the feed section. The extruded strands were then air-dried and pelletized. The pellet were then moved to hot press machine at Csic laboratory in The University of Sheffield for sheet fabrication. Parameter of hot press machine was 10 minutes of pre-heating followed by 10 minutes of hot pressing under 5 bar of pressure. The mould was taken out from machine and allowed to cool down in ambient temperature for 10 minutes.

Table 1837. Combination formula.

No. of sample	Sample name	Floreon, wt%	Kenaf, fibrewt%	Magnesium Hydroxide, wt%
1	FLO	100	-	-
2	5KF	95	5	-
3	10KF	90	10	-
4	5MH	95	-	5
5	10MH	90	-	10
6	5KF5MH	90	5	5
7	5KF10MH	85	5	10
8	10KF5MH	85	10	5
9	10KF10MH	80	10	10

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Scanning electron microscopy (SEM)

The morphology of specimen was studied by using scanning electron microscopy (SEM) according to ASTM D 6045. The testing specimens were obtained from flexural testing specimens, to study the bending failure mechanism. Image of cross-sections of specimens were carried out under accelerating voltage of 15kV.

Thermogravimetry Analysis (TGA)

TGA measurement was performed under nitrogen flow from ambient temperature to 600 ^{o}C by a rate of 30 ^{o}C/min in Chemical Engineering Department at Universiti Putra Malaysia (UPM). The weight of the sample used was roughly 10 mg and performed by technician in Chemical Engineering Department.

Differential Scanning Calorimetry (DSC)

DSC measurement was performed at the Csic laboratory in University of Sheffield with a Perkin Elmer DSC 8000 Advanced Double-Furnace. The crystallization temperature (T_c), the melting temperature (T_m) and the glass transition temperature (T_g) were evaluated in the testing. Approximately 5 mg of the samples were sealed in an aluminium plate. The testing was started at ambient temperature and raised up to 200 ^{o}Cat a rate of 10 ^{o}C/min (Sacchetin et al., 2016). The sample was held at 200 ^{o}C for a minute and then its temperature was decreased back to ambient temperature with a 10 ^{o}C/min cooling rate. The sample was held at ambient temperature for a minute and immediately a 2nd heating cycle was conducted up to 200 ^{o}C with same heating

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rate. The first heating cycle was used to remove the thermal history and moisture from the sample.

Dynamic Mechanical Analysis (DMA)

DMA testing under a dual cantilever mode was implemented with a sample dimension of 50 mm x 10 mm x 5 mm. The measurements were carried out in the temperature range from ambient temperature up to 200^{o}C, with a scanning rate of 2^{o}C/min at a frequency of 1Hz. The sample was clamped on both sides and the stress, σ was applied sinusoidal by a movable clamp at the centre of the clamped sample. The response from the sample was measured as strain, ε. The strain was obtained as the retarded lag of the stress applied due to the behaviour of the sample. The dynamic modulus, E', and the dynamic loss modulus, E'', were determined by the phase angle, δ, which was calculated from Eq. 2:

$$\tan\delta = E''/E' \qquad \text{Equation 2 (Idicula et al., 2005)}$$

RESULTS AND DISCUSSION

Table 19 shows the mass lost in every stage with the peak temperature as well as the residual mass at 600 ^{o}C. Sample 1 has clean one stage thermal decomposition at 377.941 ^{o}C with 99.795 % of the mass lost. Only a small amount of residual is expected and this is in has agreement with Ho (2015) and Awal (2015). Sample 2 and 3 have lower single stage decomposition temperatures due to the appearance of KF, which has lower thermal stability in nature (Hao et al., 2013;Bakare et al., 2016). However, a higher residual mass at the end of the testing was attributed to the formation of ash by the high lignin amount of KF (Deka et al., 2013). Figure 36 shows the SEM micrograph of sample 3, indicating good interfacial bonding between the fibre and the matrix, causing a higher residual mass (Ismail et al., 2011).

Table 1938. Mass lost in every stage with the peak temperature as well as residual mass at 600 °C for TGA.

Sample	1st mass lost, %	Temperature, °C	2nd mass lost, %	Temperature, °C	3rd mass lost, %	Temperature, °C	Mass Residual at 600 °C, %
1	99.795	377.941	-	-	-	-	0.205
2	98.161	363.001	-	-	-	-	1.839
3	96.781	357.408	-	-	-	-	3.219
4	80.734	298.661	8.175	336.155	6.636	450.632	4.428
5	65.680	281.002	13.440	347.475	12.630	462.705	8.240
6	74.960	285.437	12.920	335.818	6.040	450.707	6.060
7	64.500	275.205	14.380	342.238	11.150	456.669	9.970
8	71.180	280.874	15.289	342.998	6.835	451.561	6.693
9[a]	49.850	287.233	18.340	347.65	18.550	462.775	11.760

[a] mass loss of 1.99% has found at 155.171 °C was caused by evaporation of water

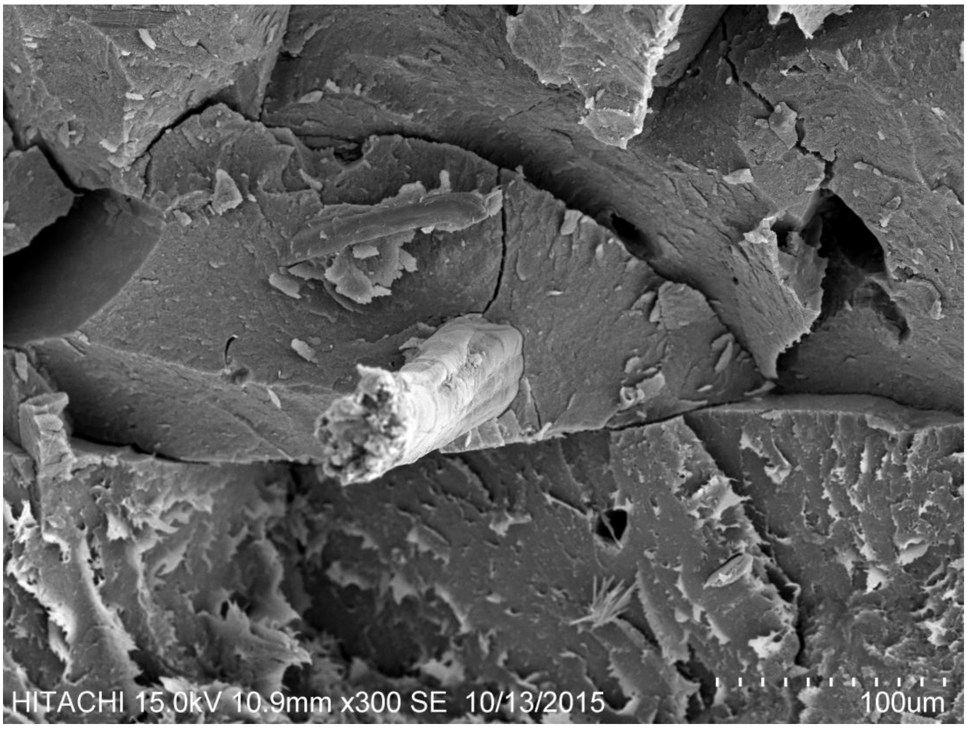

Figure 72. SEM micrograph of sample 3.

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Sample 4 and 5 have a more complex stage of thermal decomposition, caused by the MH flame retardant filler. A product of magnesium oxide and its side product, water vapour have been produced by the endothermic reaction of the MH (eq.3), which it undergoes at 332 ^{o}C. Besides, the bonding between the MH and polyester based FLO has also attributed to the complex thermal decomposition (eq.4) (Shand, 2006).

$Mg(OH)_2\ (s) > MgO\ (s) +\ H_2O\ (g)$ Equation 3 (Shand, 2006)
RCOO$^-$….Mg$^{2+\cdot}$…$^-$OOCRCOO$^-$….Mg$^{2+\cdot}$ …$^-$OOCR Equation 4 (Shand, 2006)

The heat absorbed in the reaction slow the burning process. The water vapour released dilutes the flammable gases and inhibits oxygen from the combustion. The residual mass at 600 ^{o}C was expected to be higher than the pure FLO biopolymer and the KF reinforced FLO biocomposites. Mass residuals of 4.43% and 8.24% were found in samples 4 and 5 respectively. However, MH fillers were found not to be the best flame retardant for FLO biopolymer composites, as pure FLO biopolymer has a higher decomposition temperature than the MH reaction temperature. From the results, the early start of thermal decomposition may be due to the low interface bonding between the MH filler and the FLO biopolymer. The second thermal decomposition stage was accounted for by the loss of the water vapour side product of the MH endothermic reaction. The second stage of thermal decomposition temperature was about the same as the MH endothermic reaction temperature. The last thermal decomposition stage was suspected to be due to the decomposition delayed by the released water vapour.
In the three phase biocomposites (KF/FLO/MH), the same pattern was recorded for samples 6-9. A higher KF content induced a larger portion of cellulose char and hence a higher mass residual. Higher MH loading gives the same higher mass residual due to the combustion process delay and water vapour release. This has shown some

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synergistic effect in char formation (Subasinghe and Bhattacharyya, 2014). However, MH filler was expected to be more significant in increasing the mass residual at the end of the testing. It is worth mentioning that a mass loss of 1.99% that was found in sample 9 at 155.171 ^{o}C was caused by the evaporation of water (Ismail et al., 2011). This phenomenon demonstrates the poor interfacial bonding which is in sample 9. The KF absorbed moisture from the surrounding.

Differential Scanning Calorimetry (DSC)

The thermal properties of the samples were studied by DSC analysis. DSC is a technique for determining the quantity of heat when the sample undergoes chemical or physical change. The DSC thermograms of sample 3 are shown in Figure 73, two heating cycles were found in the graph. The first heating cycle was performed in order to delete the thermal history in the sample and also to eliminate the moisture content. Therefore the T_c in the first heating scan is influenced by the water molecules and the thermal history (Ratto et al., 1995). Tm readings of the samples were recorded at about 150^{o}C. These melting temperatures were found in the range of semi-crystalline polymer (Byun et al., 2010). The result of DSC for all samples are shown in Table ***2039***20.

The T_g of the samples were shown to be decreasing for all samples compared to the pure FLO biopolymer. This is predicted as the high brittleness of the biopolymer could be found. A lower T_g shift the samples towards more flexible and leathery properties. The KF reinforced FLO biopolymer composite has reduce its T_g value with increasing the KF content in composite, which is in agreement with the findings of Rahman (Rahman et al., 2012).KF has more effecton decreasing T_g than MH fillers and three phase system composite (KF/FLO/MH).Samples 6-9 showsthe synergetic effect of

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reducing T_g. Sample 9 has reduced its T_g from 60.31 °C (sample 1) to 50.37 °C. The melting temperature was found to have no significant change when either KF or MH or both were inserted. This has agreed by Liu (2014). Besides this, the MH reacting temperature was above 330 °C, showing that MH has no influence on the melting temperature of the sample.

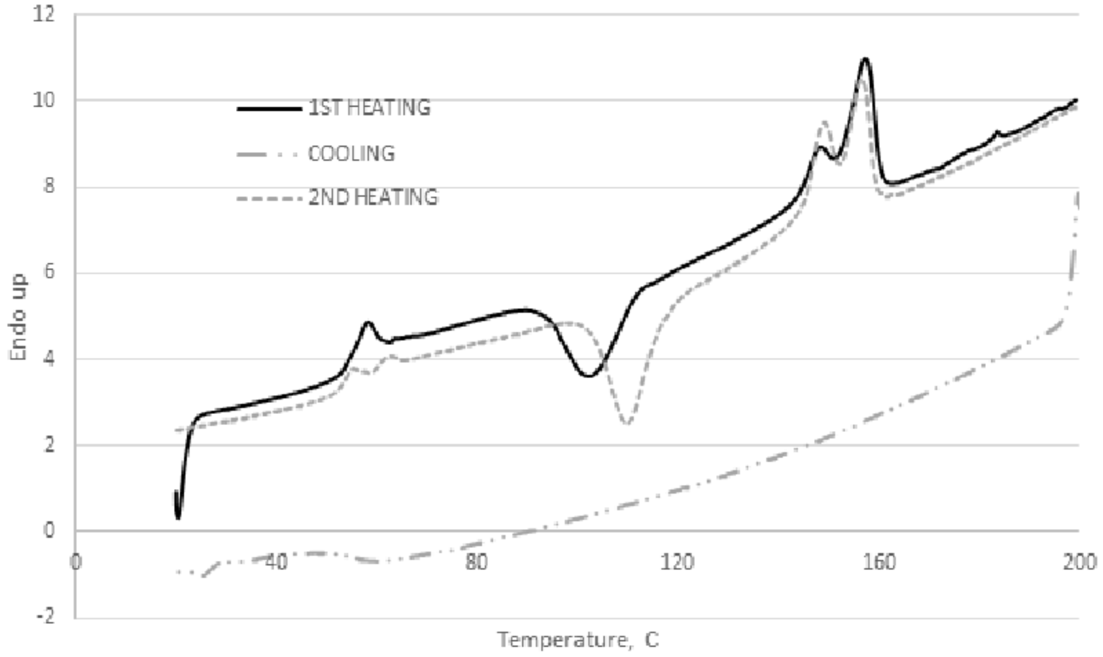

Figure 73. DSC thermograms of sample 3.

Table 2039: Result of DSC.

Sample	T_g	T_c (1st heating)	T_c (2nd heating)	T_{m1}	T_{m2}
1	60.31	107.73	115.7	148.8	155.22
2	54.01	94.23	110.62	146.12	156.07
3	51.35	102.65	109.93	148	157.34
4	54.01	109.12	116.43	148.98	156.18
5	53.51	107.55	114.89	156.56	166.47
6	53.56	93.06	115.13	146.73	157.24
7	53.04	102.18	115.78	147.58	156.7
8	52.88	102.52	114.14	147.19	156.65

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9	50.37	100	116.5	145.43	156.02

Dynamic Mechanical Analysis (DMA)

The Figure 74, Figure 75 and Figure ***4076***40 representing the graph of storage modulus, loss modulus and tan δ of all sample, respectively. The dynamic storage modulus (E') is the most important measurement to determine the load-bearing capability of the material throughout a range of temperature and it like the flexural modulus. The ratio of the loss modulus (E'') to the storage modulus (E') is known as the mechanical loss factor (tan δ). Tan δ is a quantity to measure the phase difference between the elastic and the viscous phase. Polymers are considered as viscoelastic materials, partially elastic and partially viscous. Therefore the phase differences, tan δ,is not the absolute zero phase nor the perpendicular phase. The tan δ peak is normally found in 10 – 20 ^{o}C above the T_g, which is very close to the peak temperature of the loss modulus, E''.

The effectiveness of the fillers on the moduli of the composites can be represented by a co-efficient C by the following equation (e.q.5),

$$C = \frac{(E'_G/E'_R)\ composite}{(E'_G/E'_R)\ resin}$$ Equation 5 (Idicula et al., 2005)

where E'_G and E'_R are the storage modulus values in the glassy and rubber region, respectively. The lower the value of the constant C, the higher is the effectiveness of the filler. The values of E' were taken at 45 and 100 ^{o}C to use as E'_G and E'_R respectively. The values of C recorded for the different sample are plotted in Table ***2140***21. The values decrease with the increasing fibre loading. This is agreed by

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Idicula *et al.* (2005) and Pothen *et al.*(2003). This shows that the fibres transfer the loading effectively.

The hydrophilic nature of the kenaf induces poor adhesion with polymer, and the presence of moisture promotes the voids formations (Saha et al., 1999). Therefore, slightly lower modulus recorded on sample 2, which have 5 and 10 wt% of KF but higher modulus for sample 3, compared to the neat FLO polymer. Close value of the storage moduli found in DMA for all samples except sample 4. This slightly difference of modulus have found similar from other previous work (Saha et al., 1999).

All sample composites expect sample 5 has lower T_g value, compared to neat FLO polymer. This is an unusual behaviour of filler composites like other work, rough surface of natural fibre has reported helps in initiate the crystal growth (Mathew et al., 2006). The reduction of T_g in this study were suspected to high moisture and incompatible between fillers and polymer has overcome the effect of crystal growth on fibre surface, making the chain movement easier in lower temperature.

Tan δ is a damping term that is related to impact resistance of a material. It is found that a neat polymer has a very high tan δ peak at T_g and this is similar to other work (Idicula et al., 2005). A lower tan δ peak was expected with the addition of KF and MH fillers and synergetic effect found in three phase system (sample 7-9). This is because of the lower volume fraction of the polymer caused by inserting KF and/or MH fillers, hence lower polymer chain mobility (Nyambo et al., 2010;Sreekumar et al., 2010). This finding is consistent with the co-efficient C value, meaning the effective load transfer mechanism. The SEM micrographs of the flexural fracture surfaces of sample 7 are shown in Figure 30(b). It is noticed that good interfacial bonding was found in the sample 7.

Table 2140. DMA result.

Sample	T_g	tan δ peak	C
1	55.63	1.95	-
2	50.96	1.5	0.52
3	53.42	0.84	0.21
4	51.61	0.74	0.18
5	57.54	1.38	0.46
6	53.78	1.17	0.29
7	49.23	0.22	0.12
8	50.18	0.88	0.19
9	50.8	0.68	0.12

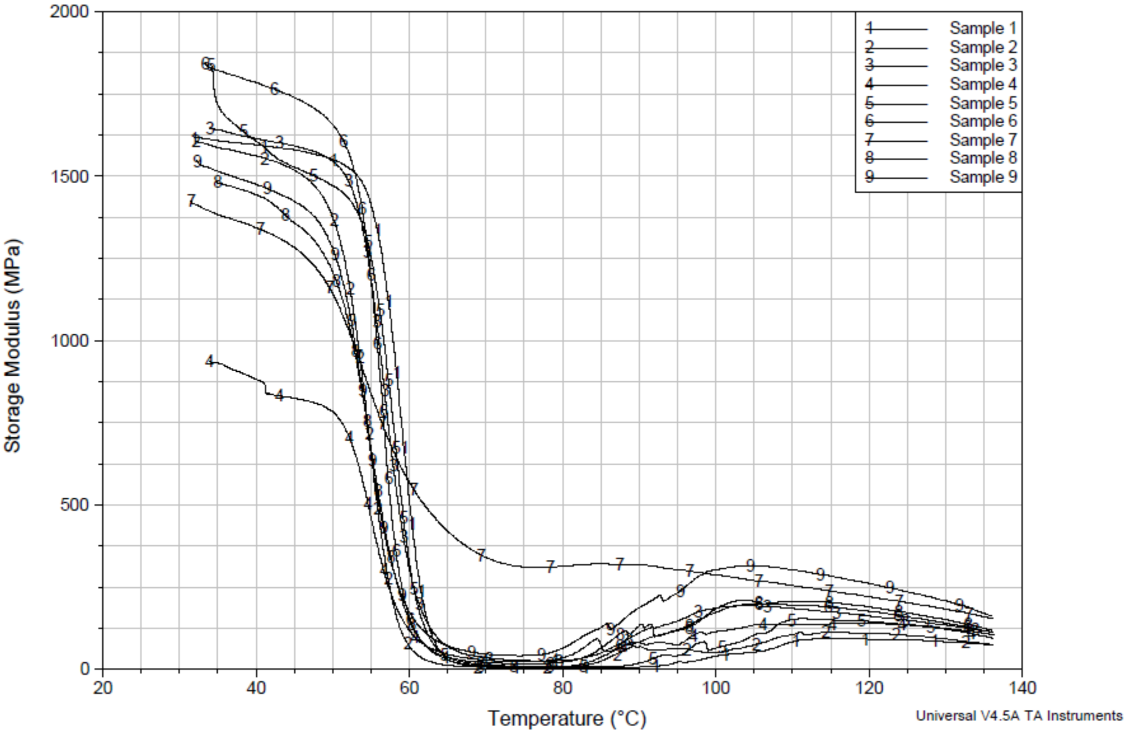

Figure 74. Storage modulus of samples.

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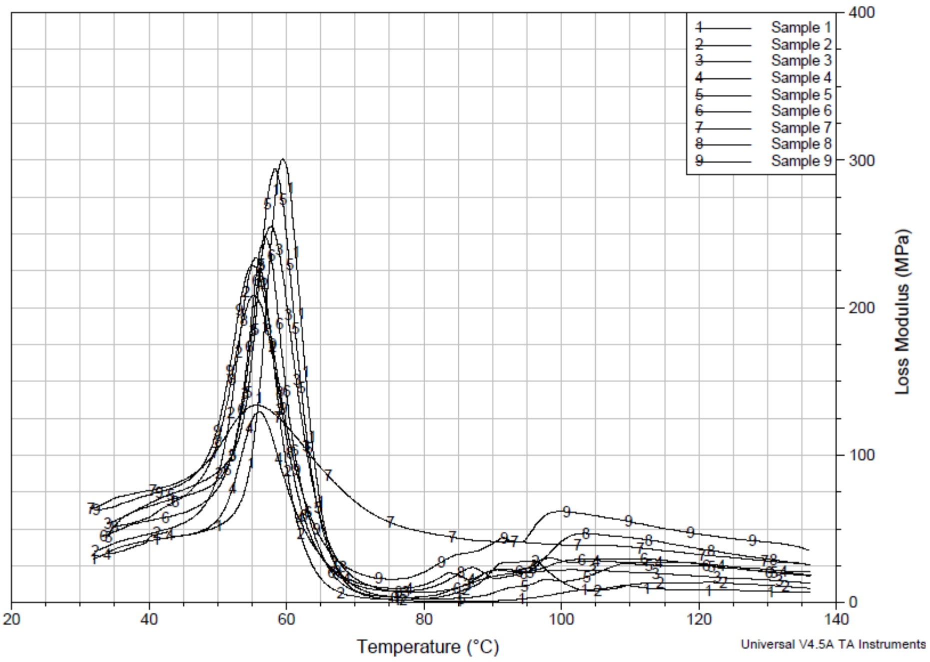

Figure 75. Loss modulus of samples.

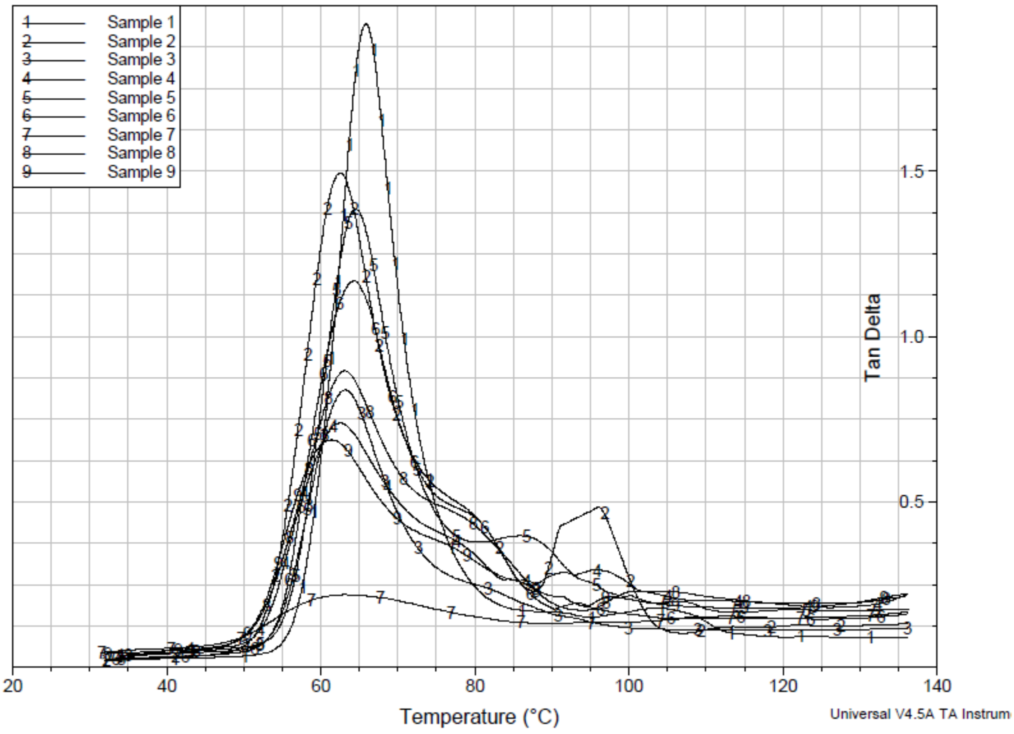

Figure 4076. Tan δ of samples.

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CONCLUSIONS

As conclusion, insertion of the kenaf fibre (KF) and magnesium hydroxide (MH) have influenced the properties of composites. The thermal instability of natural fibre has found a lower decomposition temperature for the KF reinforced composite but a higher residual mass at 600 ^{o}C. A product of magnesium oxide and its side product, water vapour, have produced in an endothermic reaction of the MH fillers undergoes at 332 ^{o}C, causing complex stage of decomposition. The water vapour released diluted the flammable gases and inhibits oxygen from the combustion. The MH filler contained composite has higher residual mass at 600 ^{o}C than the pure FLO biopolymer and the KF reinforced FLO biocomposites. However, MH fillers found not the best flame retardant for the FLO biopolymer composites as pure FLO biopolymer has higher decomposition temperature than MH reaction temperature. Some synergistic effect in char formation, T_g reduction and a lower tan δ peak shown in the three phase's biocomposites (KF/FLO/MH). MH filler is expected having more impactfor increasing mass residual. The T_g of sample was show decreasing for all samples compared to the pure FLO biopolymer. The melting temperature has found no significant change either KF or MH or both of these were inserted. MH reacting temperature was above 330 ^{o}C, saying that the MH has no influences in melting temperature of sample. The lower volume fraction of polymer by inserting the KF and/or MH fillers, hence lower polymer chain mobility. The values of co-coefficient, C recorded decreasing as increasing the fibre loading. This showing the fibres transfer the loading effectively. Close value of the storage moduli found in DMA for all samples except sample 4.

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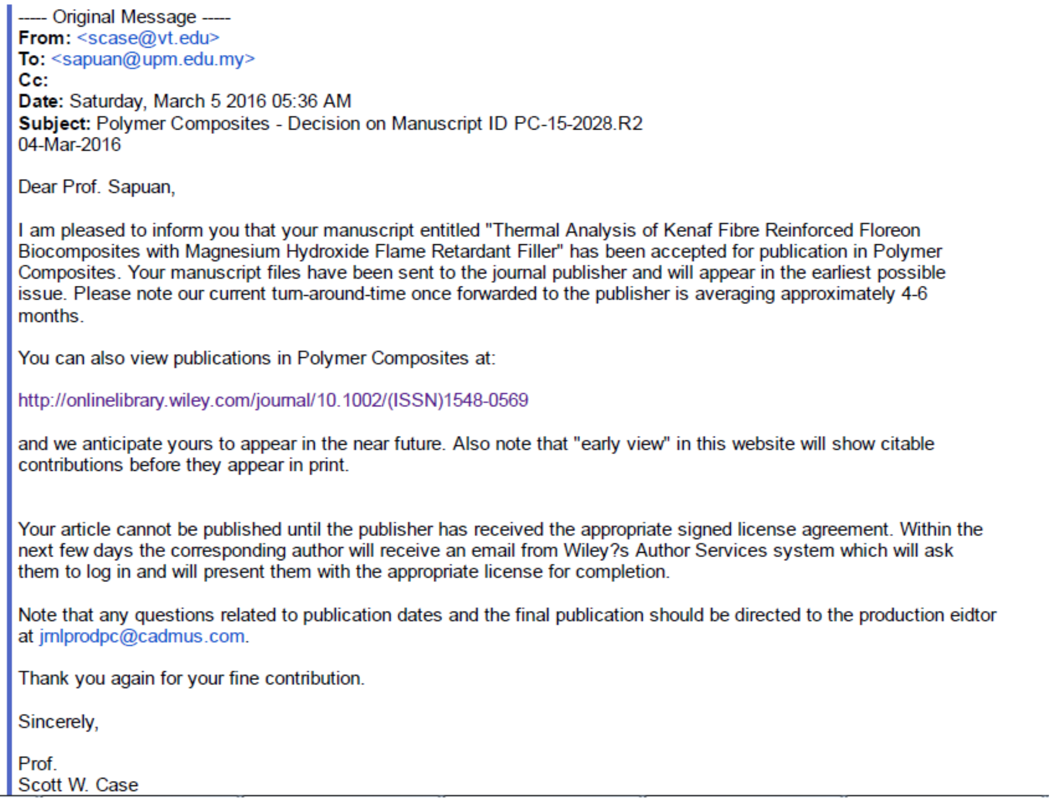
Acceptance email



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CHAPTER 8

RESULTS AND DISCUSSION

KF reinforced FLO composites with MH flame retardant is a potential biocomposites that can be utilized to develop environmentally friendly aircraft interior panel material to reduce fire incident and extend escaping time when air craft fire incident. Besides, the development of such fully biodegradable composite material is instrumental in the effort to address the current environmental issues created by the disposal of non-biodegradable plastics. Unfortunately, the limitations of KF reinforced FLO polymer composites, such as low fire-retardant properties, has limit their wide application in hazard advanced industrial. The enhancement of the thermal properties of KF reinforced FLO composites was the challenge undertaken in this current study. Hence two modification techniques were employed in this study: (1) incorporated KF into PP polymer with MH flame retardant and (2) reinforces KF and MH by different combination of volume. The sample specimens have been prepared for different volume contents of KF and MH in PP and FLO polymer, by using extrusion followed by hot pressing method. The results from both types of KF polymer composites have shown better strength properties for PP composites compared to FLO polymer. Besides, TGA testing have recorded no significant differences on second degradation temperature for both MH contented composite, yet better mass residual and first decomposition temperature have noted for PP polymer composites. Although statistical results have proof better performances on several perspectives for KF reinforced PP composites with MH flame retardant fillers, environmental problem is one thing we cannot ignored. FLO biopolymer composite is definitely a fully biodegradable composite. Therefore, melt flow properties of the bio-composites have studied for commercial production intention. Suitable melt flow properties will help

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better surface finish and save time and cost. The increment of the melt flow properties (MVR and MFR) was found for the KF or MH insertion, due to the hydrolytic degradation of the polylactic acid (PLA) in FLO. Complicated melt flow behaviour of the KF reinforced FLO/MH biocomposites was recorded. High probability of KF-KF and KF-MH collisions was expected and more collisions for higher fibre and filler loading, causing lower melt flow properties.

On the other hand, insufficient resin for fibre wetting, hydrolytic degradation and poor interfacial bonding were attributed to low strength profile. Nevertheless, inserting KF and MH filler have shown positive outcome on flexural modulus but deterioration on impact strength. Further addition of KF was recorded increases in tensile, flexural and impact strength. Meanwhile, FLO is a hydrophobic biopolymer. In this regard, for the first 24 hours, the water absorption rates were high for all bio-composites except FLO polymer. Hence, it is worth mentioning that the high contents of KF in bio-composites shown higher saturation period and higher total amount of water absorption while MH caused shorter saturation period but lower total amount of water absorption. Besides, interface bonding incompatibility has increased the water absorption of KF/FLO/MH composites.

Synergistic effects were found for char formation, T_g reduction and a lower tan δ peak in the three-phase system (KF/FLO/MH). The MH filler was found to be more significant in enhancing mass residual. The T_g were show deterioration for all samples compared to pure FLO biopolymer. The melting temperature has found no meaningful change for either insertion of KF or MH or both. As conclusion, although 10KF5MH specimen does not have the best performance in mechanical properties, a higher flame retardancy shall provide KF reinforced FLO composite with MH filler for more applications in advanced sector especially, in hazardous environment.

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CHAPTER 9

CONCLUSION AND FUTURE RESEARCH

9.1 Conclusion

As the like aircraft exterior, interiors have been put on a weight-loss scheme. Every hundreds of kilogram reduction will save about 1% of fuel consumption. However, Fire behaviour properties are significant factors for natural fibre based polymer composites in terms of safety. Understanding its flammability factors and improving them will help to delay passenger panic and hence extend the escape time.

In this study, all specific objectives have been achieved. All the sample have been fabricated with different volume contents of kenaf fibre added into polypropylene and Floreon polymer. This is to find out the optimum combination for good fire retardancy behaviour as well as maintaining good strength for kenaf fibre composites. Although better mechanical and thermal properties have been found for kenaf fibre reinforced polypropylene composite. Non-biodegradable polymer polypropylene has caused tonnes of the landfill issues in previous decade. Therefore, biocomposite will be considered. It has been concluded that best Floreon biocomposite in study was 10 wt% of kenaf fibre reinforced Floreon biocomposite with 5wt% magnesium hydroxide filler and it has reduce its first mass loss to 71.18% instead of 99.8% for pure polymer. Besides, lower glass transition temperature from the results of DSC and DMA have proof the sample become more ductile with higher modulus strength value. It is also showing best mechanical properties among all the three-phase system composite (Fibre-polymer-filler). A part of this, higher melt flow rate of the kenaf fibre reinforced Floreon composite with magnesium hydroxide additives found in this study.

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Higher flow rate enables smooth flow of the polymer and hence better fibre wetting and lower fabrication difficulties.

9.2 Recommendations for future research

- The other fibre pre-treatment such as silane treatment can be evaluated for properties of KF reinforced FLO composites
- Ammonium polyphosphate, clay particles can be applied for KF reinforced FLO composites to enhance its fire retardancy.
- The performance of the composites in more perspectives can be studied by using different testing such as horizontal burning test, smoke component test and TGA in oxygen condition.

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REFERENCES

Abdul Rashid, A. M.&L. T. Chew (1990). Fire retardant treated chipboards. *Proceeding Conferrenc on Forestry and Forest Product Research, CFFPR, Forest Research Institute Malaysia*.

Aero, B. (2001). "Flight and cabin crew response to in-flight smoke." 2013, from www.boeing.com/commerial/aeromagazine/aero_14/inflight_story.html.

Aggarwal, P. K., N. Raghu, A. Karmarkar&S. Chuahan (2013). "Jute–polypropylene composites using m-TMI-grafted-polypropylene as a coupling agent." *Materials & Design 43*: 112-117.

Aimi, N. N., H. Anuar, M. R. Manshor, W. B. W. Nazri&S. M. Sapuan (2014). "Optimizing the parameters in durian skin fibre reinforced polypropylene composites by response surface methodology." *Industrial Crops and Products 54*: 291-295.

Aisyah, H. A., M. T. Paridah, M. H. Sahri, U. M. K. Anwar&A. A. Astimar (2013). "Properties of medium density fibreboard (MDF) from kenaf (Hibiscus cannabinus L.) core as function of refining conditions." *Composites Part B: Engineering 44(1)*: 592-596.

Akil, H. M., M. F. Omar, A. A. M. Mazuki, S. Safiee, Z. A. M. Ishak&A. Abu Bakar (2011). "Kenaf fibre reinforced composites: A review." *Materials & Design 32(8–9)*: 4107-4121.

Al-Oqla, F. M., S. M. Sapuan, T. Anwer, M. Jawaid&M. E. Hoque (2015). "Natural fibre reinforced conductive polymer composites as functional materials: A review." *Synthetic Metals 206*: 42-54.

Alavudeen, A., N. Rajini, S. Karthikeyan, M. Thiruchitrambalam&N. Venkateshwaren (2015). "Mechanical properties of banana/kenaf fibre-reinforced hybrid polyester composites: Effect of woven fabric and random orientation." *Materials & Design (1980-2015) 66, Part A*: 246-257.

Alireza A., J. H. a. M. N. M. Y. (2003). Pulping and papermaking properties of Malaysian cultivated kenaf (Hibiscus cannabinus). *Proceeding of the second technical review meeting on the national project kenaf research project, Serdang*.

Alvarez, V. A., A. N. Fraga&A. Vázquez (2004). "Effects of the moisture and fibre content on the mechanical properties of biodegradable polymer–sisal fibre biocomposites." *Journal of Applied Polymer Science 91(6)*: 4007-4016.

Amato, E., Y. A. Diaz-Fernandez, A. Taglietti, P. Pallavicini, L. Pasotti, L. Cucca, C. Milanese, P. Grisoli, C. Dacarro, J. M. Fernandez-Hechavarria&V. Necchi (2011). "Synthesis, Characterization and Antibacterial Activity against Gram Positive and Gram Negative Bacteria of Biomimetically Coated Silver Nanoparticles." *Langmuir 27(15)*: 9165-9173.

Amini, M., M. Arami, N. M. Mahmoodi&A. Akbari (2011). "Dye removal from colored textile wastewater using acrylic grafted nanomembrane." *Desalination 267(1)*: 107-113.

Amran, M., R. Izamshah, M. Hadzley, M. Shahir, M. Amri, M. Sanusi&H. Hilmi (2015). "The Effect of Binder on Mechanical Properties of Kenaf Fibre/Polypropylene Composites Using Full Factorial Method." *Applied Mechanics and Materials 695*: 709-712.

Anbukarasi, K.&S. Kalaiselvam (2015). "Study of effect of fibre volume and dimension on mechanical, thermal, and water absorption behaviour of luffa reinforced epoxy composites." *Materials & Design (1980-2015) 66, Part A*: 321-330.

Anuar, H., Z. Ahmad&F. F. (2010). Biodegradable PLA-Kenaf fibre biocomposite for cleaner environment. *Malaysian Science and Technology Congress, Crystal Crown Hotel*.

Anuar, H., N.A.Hassan&F. M. Fauzey (2011). "Compatibilized PP/EPDM-Kenaf Fibre Composite using Melt Blending Method." *Advanced Materials Research 264-265*: 743-747.

Anuar, H.&A. Zuraida (2011). "Improvement in mechanical properties of reinforced thermoplastic elastomer composite with kenaf bast fibre." *Composites Part B: Engineering 42(3)*: 462-465.

Anwar, U. M. K., M. T. Paridah, H. Hamdan, S. M. Sapuan&E. S. Bakar (2009). "Effect of curing time on physical and mechanical properties of phenolic-treated bamboo strips." *Industrial Crops and Products 29(1)*: 214-219

ASTM D570-98(2010)e1. (2010). Standard Test Method for Water Absorption of Plastics. *West Conshohocken, PA, ASTM International*.

ASTM D6110-10. (2010). Standard Test Method for Determining the Charpy Impact Resistance of Notched Specimens of Plastics. *West Conshohocken, PA, ASTM International*.

ASTM D638-14. (2014). Standard Test Method for Tensile Properties of Plastics. *West Conshohocken, PA, ASTM International*

Asumani, O. M. L., R. G. Reid&R. Paskaramoorthy (2012). "The effects of alkali–silane treatment on the tensile and flexural properties of short fibre non-woven kenaf reinforced polypropylene composites." *Composites Part A: Applied Science and Manufacturing 43(9)*: 1431-1440.

Ausias, G., A. Bourmaud, G. Coroller&C. Baley (2013). "Study of the fibre morphology stability in polypropylene-flax composites." *Polymer Degradation and Stability 98(6)*: 1216-1224.

Awal, A., M. Rana&M. Sain (2015). "Thermorheological and mechanical properties of cellulose reinforced PLA bio-composites." *Mechanics of Materials 80, Part A*: 87-95.

Aziz, A. A. A., S. M. Alauddin, R. M. Salleh&M. Sabet (2012). "Influence of Magnesium Hydroxide/Aluminum Tri-Hydroxide Particle Size on Polymer Flame Retardancy: An Overview." *International Journal of Chemical Engineering and Applications 3(6)*: 437-440.

Azwa, Z. N., B. F. Yousif, A. C. Manalo&W. Karunasena (2013). "A review on the degradability of polymeric composites based on natural fibres." *Materials & Design 47*: 424-442.

Baiardo, M., E. Zini&M. Scandola (2004). "Flax fibre–polyester composites." *Composites Part A: Applied Science and Manufacturing 35(6)*: 703-710.

Baillie, C. (2005). Green Composites: Polymer Composites and the Environment*, Taylor & Francis.*

Baimark, Y.&P. Srihanam (2015). "Influence of chain extender on thermal properties and melt flow index of stereocomplex PLA." *Polymer Testing 45*: 52-57.

Bajpai, P. K., I. Singh&J. Madaan (2012). "Comparative studies of mechanical and morphological properties of polylactic acid and polypropylene based natural fibre composites." *Journal of Reinforced Plastics and Composites 31(24)*: 1712-1724.

Bakare, F. O., S. K. Ramamoorthy, D. Åkesson&M. Skrifvars (2016). "Thermomechanical properties of bio-based composites made from a lactic acid thermoset resin and flax and flax/basalt fibre reinforcements." *Composites Part A: Applied Science and Manufacturing 83*: 176-184.

Balakrishnan, H., H. A., I. N. A.&K. C. (2012). "On the use of magnesium hydroxide towards halogen-free flame-retarded polyamide-6/polypropylene blends." *Polymer Degradation and Stability 97*: 1447-1457.

Bernard, M., A. Khalina, A. Ali, R. Janius, M. Faizal, K. S. Hasnah&A. B. Sanuddin (2011). "The effect of processing parameters on the mechanical properties of kenaf fibre plastic composite." *Materials & Design 32(2)*: 1039-1043.

Bioplastics, E. (2011). " Feedstock recovery of post industrial and post consumer polylactide bioplastics." 2015, from http://en.european-bioplastics.org/wp-content/uploads/2011/04/fs/FactSheet_Feedstock_Recovery.pdf.

Bledzki, A. K., P. Franciszczak, Z. Osman&M. Elbadawi (2015). "Polypropylene biocomposites reinforced with softwood, abaca, jute, and kenaf fibres." *Industrial Crops and Products 70*: 91-99.

Bledzki, A. K., V. E. Sperber&O. Faruk (2002). Natural and Wood Fibre Reinforcement in Polymers, *Rapra Technology Limited.*

Blog, T. F. d. (2013) "Introducing Floreon…." Nov 15, 2015, from https://floreonblog.wordpress.com/2013/11/; .

Board, N. T. S. (1986). "Air Canada Flight 797 McDonnell Douglas DC-9-32, C-FTLU." 2015, from http://www.ntsb.gov/investigations/accidentreports/pages/AAR8602.aspx.

Board, N. T. S. (2000). "Inflight breakup over the Atlantic Ocean, Trans World Airline flight 800, Boeing 747-131, N93119." 2015,

Boeing (2015). "Statistical Summary of Commercial Jet Airplane Accidents Worldwide Operations | 1959–2015." 2016, from http://www.boeing.com/resources/boeingdotcom/company/about_bca/pdf/statsum.pdf.

Bonino, M. J. (2003). Material Properties of Spider Silk *University of Rochester Rochester, NY*

Borysiak, S., D. Paukszta&M. Helwig (2006). "Flammability of wood–polypropylene composites." *Polymer Degradation and Stability 91(12)*: 3339-3343.

Brown, W. P. (2012). "Organic stereoisomerism – stereoregular poly(propene) case study, Organic Chemistry Part 14: Isomerism Index." 2013, from http://www.docbrown.info/page07/isomerism2d.htm.

Byun, Y., Y. T. Kim&S. Whiteside (2010). "Characterization of an antioxidant polylactic acid (PLA) film prepared with -tocopherol, BHT and polyethylene glycol using film cast extruder." *J. Food Eng 100*: 239–244.

Carrot, C., B. Olalla&R. Fulchiron (2012). "Relaxation of loose agglomerates of magnesium hydroxide in a polymer melt." *Polymer 53(24)*: 5560-5567.

Cerqueira, E. F., C. A. R. P. Baptista&D. R. Mulinari (2011). "Mechanical behaviour of polypropylene reinforced sugarcane bagasse fibres composites." *Procedia Engineering 10*: 2046-2051.

Charlet, K., J.-P. Jernot, J. Breard&M. Gomina (2010). "Scattering of morphological and mechanical properties of flax fibres." *Industrial Crops and Products 32(3)*: 220-224.

Chaturvedi, A. K., D. R. Smith&D. V. Canfield (2001). "Blood carbon monoxide and hydrogen cyanide concentrations in the fatalities of fire and non-fire associated civil aviation accidents, 1991-1998." *Forensic Sci Int. 121(4)*: 183-188

Chawla, K. K. (2005). Fibrous Materials, *Cambridge University Press,University ofAlabama, Birmingham.*

Cho, D., Y. Woo&K. Lee (2014). Flame retardant kenaf/PLA biocomposites: Effect of ammonium polyphosphate. The 19th International Conference on Composite Materials

Colomba, D. B., B. Carmen&G. Antonio (2007). "Effects of Diammonium Phosphate on the Yields and Composition of Products from Wood Pyrolysis." *nd. Eng. Chem. Res. 46(2)*: 430–438.

Crowson, R. J., M. J. Folkes&P. F. Bright (1980). "Rheology of short glass fibre-reinforced thermoplastics and its application to injection molding I. Fibre motion and viscosity measurement." *Polymer Engineering & Science 20(14)*: 925-933.

D638-14, A. (2014). Standard Test Method for Tensile Properties of Plastics. *West Conshohocken, PA, ASTM International*.

D790-10, A. (2010). Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials. *West Conshohocken, PA, ASTM International*.

Datta, J.&P. Kopczyńska (2015). "Effect of kenaf fibre modification on morphology and mechanical properties of thermoplastic polyurethane materials." *Industrial Crops and Products 74*: 566-576.

Davoodi, M. M., S. M. Sapuan, D. Ahmad, A. Aidy, A. Khalina&M. Jonoobi (2011). "Concept selection of car bumper beam with developed hybrid bio-composite material." *Materials & Design 32(10)*: 4857-4865.

Davoodi, M. M., S. M. Sapuan, D. Ahmad, A. Ali, A. Khalina&M. Jonoobi (2010). "Mechanical properties of hybrid kenaf/glass reinforced epoxy composite for passenger car bumper beam." *Materials & Design 31(10)*: 4927-4932.

De Britto, D.&S. P. Campana-Filho (2004). "A kinetic study on the thermal degradation of N,N,N-trimethylchitosan." *Polymer Degradation and Stability 84(2)*: 353-361.

Degée, P., P. Dubois, R. Jérŏme, S. Jacobsen&H.-G. Fritz (1999). "New catalysis for fast bulk ring-opening polymerization of lactide monomers." *Macromolecular Symposia 144(1)*: 289-302.

Deka, H., M. Misra&A. Mohanty (2013). "Renewable resource based “all green composites” from kenaf biofibre and poly(furfuryl alcohol) bioresin." *Industrial Crops and Products 41*: 94-101.

Dietz, A. G. H. (1963). Fibrous composite materials. *International Science and Technology*.

Dillingham, G. (2011). AVIATION SAFETY: Status of FAA's Actions to Oversee the Safety of Composite Airplanes. From http://www.gao.gov/products/GAO-11-849.

Doumbia, A. S., M. Castro, D. Jouannet, A. Kervoëlen, T. Falher, L. Cauret&A. Bourmaud (2015). "Flax/polypropylene composites for lightened structures: Multiscale analysis of process and fibre parameters." *Materials & Design 87*: 331-341.

Duc, A. L., B. Vergnes&T. Budtova (2011). "Polypropylene/natural fibres composites: Analysis of fibre dimensions after compounding and observations of fibre rupture by rheo-optics." *Composites Part A: Applied Science and Manufacturing 42(11)*: 1727-1737.

El-Abbassi, F. E., M. Assarar, R. Ayad&N. Lamdouar (2015). "Effect of alkali treatment on Alfa fibre as reinforcement for polypropylene based eco-composites: Mechanical behaviour and water ageing." *Composite Structures 133*: 451-457.

El-Shekeil, Y. A., M. S. Salit, K. Abdan&E. S. Zainudin (2011). DEVELOPMENT OF A NEW KENAF BAST FIBRE-REINFORCED THERMOPLASTIC POLYURETHANE COMPOSITE.

El-Shekeil, Y. A., S. M. Sapuan, K. Abdan&E. S. Zainudin (2012). "Influence of fibre content on the mechanical and thermal properties of Kenaf fibre reinforced thermoplastic polyurethane composites." *Materials & Design 40*: 299-303.

El-Shekeil, Y. A., S. M. Sapuan&M. W. Algrafi (2014). "Effect of fibre loading on mechanical and morphological properties of cocoa pod husk fibres reinforced thermoplastic polyurethane composites." *Materials & Design 64*: 330-333.

Elfehri Borchani, K., C. Carrot&M. Jaziri (2015). "Biocomposites of Alfa fibres dispersed in the Mater-Bi® type bioplastic: Morphology, mechanical and thermal properties." *Composites Part A: Applied Science and Manufacturing 78*: 371-379.

Elsaid, A., M. Dawood, R. Seracino&C. Bobko (2011). "Mechanical properties of kenaf fibre reinforced concrete." *Construction and Building Materials 25(4)*: 1991-2001.

Ersoy, S.&M. Taşdemir (2012). "Zinc oxide (ZnO), magnesium hydroxide [Mg(OH)2] and calcium carbonate (CaCO3) filled HDPE polymer composites: Mechanical, thermal and morphological properties. ." *Fen Bilimleri Dergisi, 24(4)*: 93-104.

ERSOY, S.&M. TAŞDEMİR (2012). "Zinc oxide (ZnO), magnesium hydroxide [Mg(OH)2] and calcium carbonate (CaCO3) filled HDPE polymer composites: Mechanical, thermal and morphological properties." *MÜ Fen Bilimleri Dergisi 24(4)*: 93-104.

Etaati, A., S. Pather, Z. Fang&H. Wang (2014). "The study of fibre/matrix bond strength in short hemp polypropylene composites from dynamic mechanical analysis." *Composites Part B: Engineering 62*: 19-28.

Etaati, A., H. Wang, S. Pather, Z. Yan&S. Abdanan Mehdizadeh (2013). "3D X-ray microtomography study on fibre breakage in noil hemp fibre reinforced polypropylene composites." *Composites Part B: Engineering 50*: 239-246.

Fairuz, A. M., S. M. Sapuan, E. S. Zainudin&C. N. A. Jaafar (2016). "Effect of filler loading on mechanical properties of pultruded kenaf fibre reinforced vinyl ester composites." *Journal of Mechanical Engineering and Sciences 10(1)*: 1931-1942.

Fang, Q.&M. A. Hanna (1999). "Rheological properties of amorphous and semicrystalline polylactic acid polymers." *Industrial Crops and Products 10(1)*: 47-53.

Floreon (2009). " 3D print show. ." 2015, from http://floreon.com/resources/articles/3d-print-show.

Foundation, F. S. (2016). "ASN Aviation Safety Database." 2016, from www.aviation-safety.net/database/.

Galea, E. R.&N. C. Markatos (1987). "A review of mathematical modelling of aircraft cabin fires." *Applied Mathematical Modelling 11(3)*: 162-176.

Garlotta, D. (2001) "A Literature Review of Poly(Lactic Acid)." *Journal of Polymers and the Environment 9(2)*: 63-84.

Gašparovič, L., Z. Koreňová&J. L. (2009). Kinetic study of wood chips decomposition by TGA. 36th International Conference of SSCHE. *Tatransk´e Matliare, Slovakia,*.

Gay, D., S. V. Hoa&W. T. Stephen (2003). Composite materials, interest and properties. *Composite Material: Design and Application. US, CRC Press LLC.* **4**.

George, G., K. Joseph, E. R. Nagarajan, E. Tomlal Jose&M. Skrifvars (2013). "Thermal, calorimetric and crystallisation behaviour of polypropylene/jute yarn bio-composites fabricated by commingling technique." *Composites Part A: Applied Science and Manufacturing 48*: 110-120.

Giancaspro, J., C. Papakonstantinou&P. Balaguru (2009). "Mechanical behavior of fire-resistant biocomposite." *Composites Part B: Engineering 40(3)*: 206-211.

Gibson, A. G.&A. P. Mouritz (2006). Fire Properties of Polymer Composite Materials. *Dordrecht, The Netherlands, Springer.*

Gilbert, R. D., V. Stannett, C. G. Pitt&A. Schindler (1982). "The design of biodegradable polymers: two approaches." *Polymer Degradation and Stability 4*: 259-293.

Glasbrenner, B. (2005). "NatureWorks® PLA – Commercial Development of Biopolymers on a World Scale." Retrieved 29 March, 2015,

Gorrasi, G.&R. Pantani (2013). "Effect of PLA grades and morphologies on hydrolytic degradation at composting temperature: Assessment of structural modification and kinetic parameters." *Polymer Degradation and Stability 98(5)*: 1006-1014.

Gu, H. (2009). "Dynamic mechanical analysis of the seawater treated glass/polyester composites." *Materials & Design 30(7)*: 2774-2777.

Hall, G. S. (1975). Flame retardant Treatments- The state of art,. *Record of 1975 Annual Convetion of BWPA:* 83 – 95.

Hammes, W. P.&C. Hertel (2006). The genera Lactobaccilus and Carnobacterium. *The Prokaryotes, Springer, New York.*

Han, S. O., M. Karevan, I. N. Sim, M. A. Bhuiyan, Y. H. Jang, J. Ghaffar&K. Kalaitzidou (2012). "Understanding the Reinforcing Mechanisms in Kenaf Fibre/PLA and Kenaf Fibre/PP Composites: A Comparative Study." *International Journal of Polymer Science 2012*: 8.

Hao, A., H. Zhao&J. Y. Chen (2013). "Kenaf/polypropylene nonwoven composites: The influence of manufacturing conditions on mechanical, thermal, and acoustical performance." *Composites Part B: Engineering 54*: 44-51.

Hartmann, M. H. (1998). High Molecular Weight Polylactic Acid Polymers. *Biopolymers from Renewable Resources. D. L. Kaplan. Berlin, Heidelberg, Springer Berlin Heidelberg*: 367-411.

Hassan, A., A. A. Salema, F. N. Ani&A. A. Bakar (2010). "A review on oil palm empty fruit bunch fibre-reinforced polymer composite materials." *Polymer Composites 31(12)*: 2079-2101.

Hassan, M. S. (2015). "Removal of reactive dyes from textile wastewater by immobilized chitosan upon grafted Jute fibres with acrylic acid by gamma irradiation." *Radiation Physics and Chemistry 115*: 55-61.

Helwig, M.&D. Paukszta (2000). "Flammability of Composites Based on Polypropylene and Flax Fibres." *Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals 354(1)*: 373-380.

Helwig, M., D. Paukszta, J. Garbarczyk&S. Borysiak (2000). omposites based on polypropylene and flax fibres: A study of fire performance and some physical and mechanical properties. *Proceedings from the Third Int, Symposium on Natural Polymers and Composites ISNaPol/2000, Sao Pedro, Brazil*

Henton. D. E. (2005). Polylactic acid technology. *Natural Fibres, Biopolymers, and Biocomposites, Boca Raton: Crc Press-Taylor & Francis Group*

Herrera-Franco, P. J.&A. Valadez-González (2004). "Mechanical properties of continuous natural fibre-reinforced polymer composites." *Composites Part A:*

`

Applied Science and Manufacturing 35(3): 339-345.

Ho, M.-p., K.-t. Lau, H. Wang&D. Hui (2015). "Improvement on the properties of polylactic acid (PLA) using bamboo charcoal particles." *Composites Part B: Engineering 81*: 14-25.

Ho, M.-p., H. Wang, J.-H. Lee, C.-k. Ho, K.-t. Lau, J. Leng&D. Hui (2012). "Critical factors on manufacturing processes of natural fibre composites." *Composites Part B: Engineering 43(8)*: 3549-3562.

Horrocks, A. R.&D. Price (2001). Fire Retardant Materials*, CRC Press.*

Huang, S. J. (2005). Poly(lactic acid) and copolyesters. *Handbook of Biodegradable Polymers, Rapra Technology.*

Huda, M. S., L. T. Drzal, A. K. Mohanty&M. Misra (2006). "Chopped glass and recycled newspaper as reinforcement fibres in injection molded poly(lactic acid) (PLA) composites: A comparative study." *Composites Science and Technology 66(11–12)*: 1813-1824.

Ibrahim, M. S., S. M. Sapuan&A. A. Faieza (2012). "mechanical and thermal properties of composites from unsaturated polyester filled with oil palm ash." *Journal of Mechanical Engineering and Sciences 2*: 133-147.

Idicula, M., S. K. Malhotra, K. Joseph&S. Thomas (2005). "Dynamic mechanical analysis of randomly oriented intimately mixed short banana/sisal hybrid fibre reinforced polyester composites." *Composites Science and Technology 65(7–8)*: 1077-1087.

IRWIN, L. (1991). Asphyxiation Factor In Plane Crash Deaths Officials: Smoke Hoods Would Save Lives. Los Angeles Times. From http://articles.sun-sentinel.com/1991-02-24/features/9101100343_1_smoke-hoods-british-crash-smoke-inhalation.

Ishak, M. R., Z. Leman, S. M. Sapuan, A. M. M. Edeerozey&I. S. Othman (2010). "Mechanical properties of kenaf bast and core fibre reinforced unsaturated polyester composites." *IOP Conference Series: Materials Science and Engineering 11(1)*: 012006.

Ishak, M. R., S. M. Sapuan, Z. Leman, M. Z. A. Rahman, U. M. K. Anwar&J. P. Siregar (2013). "Sugar palm (Arenga pinnata): Its fibres, polymers and composites." *Carbohydrate Polymers 91(2)*: 699-710.

Ismail, A., A. Hassan, A. B. A.&M. Jawaid (2013). "Flame Retardancy and Mechanical Properties of Kenaf Filled Polypropylene (PP) Containing Ammonium Polyphosphate (APP)." *Sains Malaysiana 42(4)*: 429–434.

Ismail, A. E.&M. A. C. A. Aziz (2015). "Tensile strength of woven yarn kenaf fibre reinforced polyester composites." *Journal of Mechanical Engineering and Sciences 9*: 1695-1704.

Ismail, H., A. H. Abdullah&A. A. Bakar (2011). "Influence of acetylation on the tensile properties, water absorption, and thermal stability of (High-density polyethylene)/(soya powder)/(kenaf core) composites." *Journal of Vinyl and Additive Technology 17(2)*: 132-137.

Izran, K., A. Zaidon, G. Beyer, A. M. A. Rashid, F. Abood&S. Rahim (2010). "OPTIMISING TREATMENT SYSTEM FOR KENAF (HIBISCUS CANNABINUS) PARTICLEBOARD WITH FIRE RETARDANTS." *Journal of Tropical Forest Science 22(2)*: 175-183.

Izran Kamal, A. R. A. M., Mohd. Nor Mohd Yusof, Khairul Masseat, Zaidon Ashaari, Faizah Abood (2009). "Physical and Mechanical Properties of Flame Retardant-Treated Hibiscus Cannabinus Particleboard." *Modern Applied Science 3(8)*: 2-8.

Jawaid, M.&H. P. S. Abdul Khalil (2011). "Cellulosic/synthetic fibre reinforced polymer hybrid composites: A review." *Carbohydrate Polymers 86(1)*: 1-18.

Jeencham, R., N. Suppakarn&K. Jarukumjorn (2014). "Effect of flame retardants on flame retardant, mechanical, and thermal properties of sisal fibre/polypropylene composites." *Composites Part B: Engineering 56*: 249-253.

John, M. J., C. Bellmann&R. D. Anandjiwala (2010). "Kenaf–polypropylene composites: Effect of amphiphilic coupling agent on surface properties of fibres and composites." *Carbohydrate Polymers 82(3)*: 549-554.

John, M. J.&S. Thomas (2008). "Biofibres and biocomposites." *Carbohydrate Polymers 71(3)*: 343-364.

Joseph, P.&J. Ebdon (2000). Recent developments in flame-retarding thermoplastics and thermosets, Fire Retardant Materials,. *Cambridge, UK., Woodhead Publishing Limited.*

Julkapli, N. M.&H. M. Akil (2010). "Thermal Properties of Kenaf-Filled Chitosan Biocomposites." *Polymer-Plastics Technology and Engineering 49(2)*: 147-153.

Kandola, B. (2000). Nanocomposites, Fire Retardant Materials. *Cambridge, UK., Woodhead Publishing Limited.*

Karaduman, Y., M. M. A. Sayeed, L. Onal&A. Rawal (2014). "Viscoelastic properties of surface modified jute fibre/polypropylene nonwoven composites." *Composites Part B: Engineering 67*: 111-118.

Kéki, S., I. Bodnár, J. Borda, G. Deák&M. Zsuga (2001). "Melt Polycondensation of d,l-Lactic Acid: MALDI-TOF MS Investigation of the Ring-Chain Equilibrium." *The Journal of Physical Chemistry B 105(14)*: 2833-2836.

Keller, A., D. Bruggmann, A. Neff, B. Müller&E. Wintermantel (2000). "Degradation Kinetics of Biodegradable Fibre Composites." *Journal of Polymers and the Environment 8(2)*: 91-96.

Khalina, A., E. S. Zainuddin&I. S. Aji (2011). "Rheological Behaviour of Polypropylene/Kenaf Fibre Composite: Effect of Fibre Size." *Key Engineering Materials 471-472*: 513-517.

Kharas G. D., S.-R. F. a. S. D. K. (1994). Plastic from Microbes*, Hanser-Gardner, Munich.*

Kim, H.-S., H.-S. Yang&H.-J. Kim (2005). "Biodegradability and mechanical properties of agro-flour–filled polybutylene succinate biocomposites." *Journal of Applied Polymer Science 97(4)*: 1513-1521.

Kozłowski, R.&M. Władyka-Przybylak (2008). "Flammability and fire resistance of composites reinforced by natural fibres." *Polymers for Advanced Technologies 19(6)*: 446-453.

Kricheldorf, H. R., I. Kreiser-Saunders&C. Boettcher (1995). "Polylactones: 31. Sn(II)octoate-initiated polymerization of L-lactide: a mechanistic study." *Polymer 36(6)*: 1253-1259.

Ku, H., H. Wang, N. Pattarachaiyakoop&M. Trada (2011). "A review on the tensile properties of natural fibre reinforced polymer composites." *Composites Part B: Engineering 42(4)*: 856-873.

Kuchinda, N. C., W. B. Ndahi, S. T. O. Lagoke&M. K. Ahmed (2001). "The effects of nitrogen and period of weed interference on the fibre yield of kenaf (Hisbiscus cannabinus L.) in the northern Guinea Savanna of Nigeria." *Crop Protection 20(3)*: 229-235.

Kuo, W.-S., T.-H. Ko&H.-I. Chen (1998). "Elastic moduli and damage mechanisms in

3D braided composites incorporating pultruded rods." *Composites Part A: Applied Science and Manufacturing 29(5)*: 681-692.

Lafranche, E., V. M. Oliveira, C. I. Martins&P. Krawczak (2015). "Prediction of injection-moulded flax fibre reinforced polypropylene tensile properties through a micro-morphology analysis." *Journal of Composite Materials 49(1)*: 113-128.

Lam, T. T. B., K. Hori&K. Iiyama (2003). "Structural characteristics of cell walls of kenaf (Hibiscus cannabinus L.) and fixation of carbon dioxide." *Journal of Wood Science 49(3)*: 255-261.

Lee, B.-H., H.-J. Kim&W.-R. Yu (2009). "Fabrication of long and discontinuous natural fibre reinforced polypropylene biocomposites and their mechanical properties." *Fibres and Polymers 10(1)*: 83-90.

Lee, B. H., H. S. Kim, S. Lee, H. J. Kim&J. R. Dorgan (2009a). "Bio-composites of kenaf fibres in polylactide: Role of improved interfacial adhesion in the carding process." *Composites Science and Technology 69*: 2573-2579.

Lee, C. H., M. S. Salit&M. R. Hassan (2014). "A Review of the Flammability Factors of Kenaf and Allied Fibre Reinforced Polymer Composites." *Advances in Materials Science and Engineering 2014*: 8.

Lee, J. M., Z. A. Mohd Ishak, R. Mat Taib, T. T. Law&M. Z. Ahmad Thirmizir (2013). "Mechanical, Thermal and Water Absorption Properties of Kenaf-Fibre-Based Polypropylene and Poly(Butylene Succinate) Composites." *Journal of Polymers and the Environment 21(1)*: 293-302.

Leman, Z., S. M. Sapuan, M. Azwan, M. M. H. M. Ahmad&M. A. Maleque (2008). "The Effect of Environmental Treatments on Fibre Surface Properties and Tensile Strength of Sugar Palm Fibre-Reinforced Epoxy Composites." *Polymer-Plastics Technology and Engineering 47(6)*: 606-612.

Leman, Z., S. M. Sapuan, A. M. Saifol, M. A. Maleque&M. M. H. M. Ahmad (2008). "Moisture absorption behavior of sugar palm fibre reinforced epoxy composites." *Materials & Design 29(8)*: 1666-1670.

Li, B.&J. He (2004). "Investigation of mechanical property, flame retardancy and thermal degradation of LLDPE–wood-fibre composites." *Polymer Degradation and Stability 83(2)*: 241-246.

Li, S. M. (1999). "Hydrolytic degradation characteristics of aliphatic polyesters derived from lactic and glycolic acids." *Journal of Biomedical Materials Research 48*: 342-353.

Li, T. Q.&M. P. Wolcott (2004). "Rheology of HDPE–wood composites. I. Steady state shear and extensional flow." *Composites Part A: Applied Science and Manufacturing 35(3)*: 303-311.

Liang, J.-Z., C.-Y. Tang, L. Zhou, L. He&C.-P. Tsui (2011). "Melt density and flow property of PDLLA/nano-CaCO3 bio-composites." *Composites Part B: Engineering 42(7)*: 1897-1900.

Liang, J.-Z., J. Yang&C.-Y. Tang (2010). "Die-swell behavior of PP/Al(OH)3/Mg(OH)2 flame retardant composite melts." *Polymer Testing 29(5)*: 624-628.

Liang, J. Z., R. K. Y. Li&S. C. Tjong (1999). "Effects of pressure and temperature on the melt density and the melt flow rate of LDEP and glass bead-filled LDPE composite." *Journal of Materials Processing Technology 91(1–3)*: 167-171.

Liang, J. Z., R. K. Y. Li&S. C. Tjong (2000). "Effects of glass bead size and content on the viscoelasticity of filled polypropylene composites." *Polymer Testing 19(2)*: 213-220.

Liang, J. Z.&W. Peng (2009). "Melt viscosity of PP and FEP/PP blends at low shear rates." *Polymer Testing 28(4)*: 386-391.

Libolon (2015). "LIBOLON eco textiles, RePET and other polyester yarns. ." 2015, from http://www.libolon.com/eco.php.

Liodakis, S.&J. Antonopoulos (2006). Evaluating the fire retardation efficiency of diammonium phosphate, ammonium sulphate and magnesium carbonate minerals on Pistacia lentiscus L. *2006 First International Symposium on Environment Identities and Mediterranean Area*.

Liu, S.-P. (2014). "Flame retardant and mechanical properties of polyethylene/magnesium hydroxide/montmorillonite nanocomposites." *Journal of Industrial and Engineering Chemistry 20(4)*: 2401-2408.

Liu, S.-P., J.-R. Ying, X.-P. Zhou, X.-L. Xie&Y.-W. Mai (2009). "Dispersion, thermal and mechanical properties of polypropylene/magnesium hydroxide nanocomposites compatibilized by SEBS-g-MA." *Composites Science and Technology 69(11–12)*: 1873-1879.

Lyon, R. E. (1997). "Fire-Resistant Materials: Research Overview." 2015, from http://oai.dtic.mil/oai/oai?verb=getRecord&metadataPrefix=html&identifier=ADA340649.

Ma, H., Q. Zhuo&B. Wang (2009). "Electro-catalytic degradation of methylene blue wastewater assisted by Fe2O3-modified kaolin." *Chemical Engineering Journal 155(1–2)*: 248-253.

Maleque, M., F. Y. Belal&S. M. Sapuan (2007). "Mechanical properties study of pseudo-stem banana fibre reinforced epoxy composite." *The Arabian journal for science and engineering 32(2B)*: 359-364.

Maleque, M., F. Y. Belal&S. M. Sapuan (2007). "Mechanical properties study of pseudo-stem banana fibre reinforced epoxy composite." *Arabian Journal for Science and Engineering 32(2B)*: 359-364.

Maleque, M. A., F. Y. Belal&S. M. Sapuan (2007). "Mechanical properties study of Pseudo-stem Banana Fibre Reinforced Epoxy Composite." *Arabian J Sci Eng 45(1)*: 359-364.

Manshor, M. R., H. Anuar, M. N. Nur Aimi, M. I. Ahmad Fitrie, W. B. Wan Nazri, S. M. Sapuan, Y. A. El-Shekeil&M. U. Wahit (2014). "Mechanical, thermal and morphological properties of durian skin fibre reinforced PLA biocomposites." *Materials & Design 59*: 279-286.

Mansor, M. R., S. M. Sapuan, E. S. Zainudin, A. A. Nuraini&A. Hambali (2014). "Conceptual design of kenaf fibre polymer composite automotive parking brake lever using integrated TRIZ–Morphological Chart–Analytic Hierarchy Process method." *Materials & Design 54*: 473-482.

Mathew, A. P., K. Oksman&M. Sain (2006). "The effect of morphology and chemical characteristics of cellulose reinforcements on the crystallinity of polylactic acid." *Journal of Applied Polymer Science 101(1)*: 300-310.

Mazuki, A. A. M., H. M. Akil, S. Safiee, Z. A. M. Ishak&A. A. Bakar (2011). "Degradation of dynamic mechanical properties of pultruded kenaf fibre reinforced composites after immersion in various solutions." *Composites Part B: Engineering 42(1)*: 71-76.

McVeigh, T. (2010). India jet crash leaves more than 150 dead in Mangalore. theguardian.

MdRadzi, M. K. F., A. B. Sulong, N. Muhamad, M. A. MohdLatiff&N. F. Ismail (2015). "Effect of Filler Loading and NaOH Addition on Mechanical Properties of Moulded Kenaf/Polypropylene Composite." *Pertanika J. Trop.*

Agric. Sci. 38(4): 583-590.

Mehta, G., A. K. Mohanty, M. Misra&L. T. Drzal (2004) "Effect of novel sizing on the mechanical and morphological characteristics of natural fibre reinforced unsaturated polyester resin based bio-composites." *Journal of Materials Science 39(8)*: 2961-2964.

Meon, M. S., M. F. Othman, H. Husain, M. F. Remeli&M. S. M. Syawal (2012). "Improving Tensile Properties of Kenaf Fibres Treated with Sodium Hydroxide." *Procedia Engineering 41*: 1587-1592.

Mobedi, H., M. Nekoomanesh, H. Orafaei&H. Mivehchi (2006). "Studying the Degradation of Poly(L-lactide) in Presence of Magnesium Hydroxide." *IRANIAN POLYMER JOURNAL 15(1)*: 31-39.

Mohammad, N. N. B.&A. Arsad (2013). "Mechanical, thermal and morphological study of kenaf fibre reinforced rPET/ABS composites." *Malaysian Polymer Journal 8(1)*: 8-13.

Mohammed, A. A., D. Bachtiar, J. P. Siregar&M. R. M. Rejab (2016). "Effect of sodium hydroxide on the tensile properties of sugar palm fibre reinforced thermoplastic polyurethane composites." *Journal of Mechanical Engineering and Sciences 10(1)*: 1765-1777.

Mohanty, A. K., L. T. Drzal&M. Misra (2002). "Novel hybrid coupling agent as an adhesion promoter in natural fibre reinforced powder polypropylene composites." *Journal of Materials Science Letters 21(23)*: 1885-1888.

Mohanty, A. K., M. Misra&L. T. Drzal (2005). Natural Fibres, Biopolymers, and Biocomposites*, CRC Press.*

Mohanty, A. K., M. Misra&G. Hinrichsen (2000). "Biofibres, biodegradable polymers and biocomposites: An overview." *Macromolecular Materials and Engineering 276-277(1)*: 1-24.

Mwaikambo, L. Y.&M. P. Ansell (2002). "Chemical modification of hemp, sisal, jute, and kapok fibres by alkalization." *Journal of Applied Polymer Science 84(12)*: 2222-2234.

Newball, H. H.&S. A. Brahim (1976). "Respiratory response to domestic fibrous glass exposure." *Environmental Research 12(2)*: 201-207.

Nishino, T., K. Hirao, M. Kotera, K. Nakamae&H. Inagaki (2003). "Kenaf reinforced biodegradable composite." *Composites Science and Technology 63(9)*: 1281-1286.

Nor Azowa Ibrahim, Wan Md Zin Wan Yunus, M. Othman, K. Abdan&Kamarul Arifin Hadithon (2010). "Poly(Lactic Acid) (PLA)-reinforced Kenaf Bast Fibre Composites: The Effect of Triacetin." *Journal of Reinforced Plastics and Composites 29(7)*: 1099-1111.

Nyambo, C., A. K. Mohanty&M. Misra (2010). "Polylactide-based renewable green composites from agricultural residues and their hybrids." *Biomacromolecules 11*: 1654–1660.

Ochi, S. (2008). "Mechanical properties of kenaf fibres and kenaf/PLA composites." *Mechanics of Materials 40(4–5)*: 446-452.

Ophardt, C. (2003). "Addition polymers." 2013, from http://www.elmhurst.edu/~chm/vchembook/401addpolymers.html.

Osman, M. R., A. A. M. Mazuki, H. M. Akil, Z. A. M. Ishak&A. A. Bakar (2014). "Effect of chemical treatment on the mechanical properties of pultruded kenaf fibre reinforced polyester composites." *Key Eng. Mater. 594-595*: 691- 695.

Panaitescu, D. M., Z. Vuluga, M. Ghiurea, M. Iorga, C. Nicolae&R. Gabor (2015). "Influence of compatibilizing system on morphology, thermal and mechanical properties of high flow polypropylene reinforced with short hemp fibres." *Composites Part B: Engineering 69*: 286-295.

Paridah M. T., N. H. A. W., Zaidon A., Azmi I., Mohd N. M. Y. and Yuziah N. M. Y. (2009). "Bonding properties and performance of multi-layered kenaf board." *Journal of Tropical Forest Science 21(2)*: 113-122.

Parta, P. K., S. B. Warner, Y. K. Kim, P. D. Calvert&S. Adanur (2005). Nano engineered fire resistant composite fibres, NTC Project No: M02-MD08,. NTC Annual Report November 2005*:* 1-10.

Pickering, K. (2008). Properties and Performance of Natural-Fibre Composites*, Elsevier Science.*

Pilarska, A., K. Bula, K. Myszka, T. Rozmanowski, K. Szwarc-Rzepka, K. Pilarski, Ł. Chrzanowski, K. Czaczyk&T. Jesionowski (2014). Functional polypropylene composites filled with ultra-fine magnesium hydroxide. Open Chemistry. *13*.

Pothan, L. A., Z. Oommen&S. Thomas (2003). "Dynamic mechanical analysis of banana fibre reinforced polyester composites." *Composites Science and Technology 63(2)*: 283-293.

Potter, T., M. Lavado&C. Pellon (2003). "Methods for characterizing arc fault signatures in aerospace applications, Texas instruments." 2013, from www.sensata.com/download/AgingAircraftConf-Sept2003.pdf.

Premalal, H. G. B., H. Ismail&A. Baharin (2002). "Comparison of the mechanical properties of rice husk powder filled polypropylene composites with talc filled polypropylene composites." *Polymer Testing 21(7)*: 833-839.

Qu, X., A. Wirsén&A. C. Albertsson (2000). "Effect of lactic/glycolic acid side chains on the thermal degradation kinetics of chitosan derivatives." *Polymer 41(13)*: 4841-4847.

Rachini, A., G. Mougin, S. Delalande, J. Y. Charmeau, C. Barrès&E. Fleury (2012). "Hemp fibres/polypropylene composites by reactive compounding: Improvement of physical properties promoted by selective coupling chemistry." *Polymer Degradation and Stability 97(10)*: 1988-1995.

Rafatullah, M., O. Sulaiman, R. Hashim&A. Ahmad (2010). "Adsorption of methylene blue on low-cost adsorbents: A review." *Journal of Hazardous Materials 177(1–3)*: 70-80.

Rahman, S. H. A., R. A. Talib, K. Abdan, C. N. Ling, Y. A. Yusof&Y. B. Tee (2012). Thermal analysis of kenaf derived cellulose-filled poly (lactic acid) composite films. International Conference on Agricultural and Food Engineering for Life (CAFEi 2012). *Malaysia*.

Rana, A. K., B. C. Mitra&A. N. Banerjee (1997). "Short jute fibre-reinforced polypropylene composites: dynamic mechanic study." *Journal of Applied Polymer Science 71*: 531-539.

Rasal, R. M.&D. E. Hirt (2009). "Toughness decrease of PLA-PHBHHx blend films upon surface-confined photopolymerization." *J Biomed Mater Res A 88(4)*: 1079-1086.

Rashdi, A., S. Sapuan, M. Ahmad&A. Khalina (2009). "Water absorption and tensile properties of soil buried kenaf fibre reinforced unsaturated polyester composites (KFRUPC)." *Journal of Food, Agriculture and Environment 7(3-4)*: 908-911.

Rashdi, A. A. A., S. M. Sapuan, M. Ahmad&A. Khalina (2009). "Water absorption and tensile properties of soil buried kenaf fibre reinforced unsaturated polyester composites (KFRUPC)." *Journal of Food, Agriculture and Environment 7(9)*: 908-911.

Ratto, J., T. Hatakeyama&R. B. Blumstein (1995). "Differential scanning calorimetry investigation of phase transitions in water/ chitosan systems." *Polymer 36(15)*: 2915-2919.

Reid, R. G., O. M. L. Asumani&R. Paskaramoorthy (2011). The effect on the mechanical properties of kenaf fibre reinforced polypropylene resulting from alkali–silane surface treatment. 16th International conference on composite structures ICCS16. *Porto, Portugal.*

Ren, F., X. Zhang, Z. Wei, J. Chen, D. Dong, X. Li&L. Zhang (2013). "Effect of particle size and content of magnesium hydroxide on flame retardant properties of asphalt." *Journal of Applied Polymer Science 129(4)*: 2261-2272.

Ren, X., R. Qiu, L. S. Fifield, K. L. Simmons&K. Li (2012). "Effects of Surface Treatments on Mechanical Properties and Water Resistance of Kenaf Fibre-Reinforced Unsaturated Polyester Composites." *Journal of Adhesion Science and Technology 26(18-19)*: 2277-2289.

Rhim, J.-W., A. K. Mohanty, S. P. Singh&P. K. W. Ng (2006). "Effect of the processing methods on the performance of polylactide films: Thermocompression versus solvent casting." *Journal of Applied Polymer Science 101(6)*: 3736-3742.

Ridzuan, R., M. D. H. Beg, M. Y. Rosli, M. H. Rohaya, A. A. A. S. Samahani&I. Zawawi (2013). "Effects of Coupling Agent and Flame Retardant on the Performances of Oil Palm Empty Fruit Bunch Fibre Reinforced Polypropylene Composites." *International Journal of Chemical, Molecular, Nuclear, Materials and Metallurgical Engineering 7(6)*: 445-453.

Rogowski, B. F. W. (1970). The fire propagation test: Its development and application. *Fire Research Technical Paper, London*.

Rouison, D., M. Sain&M. Couturier (2004). "Resin transfer molding of natural fibre reinforced composites: cure simulation." *Composites Science and Technology 64(5)*: 629-644.

Russo, P., C. Carfagna, F. Cimino, D. Acierno&P. Persico (2013). "Biodegradable Composites Reinforced with Kenaf Fibres: Thermal, Mechanical, and Morphological Issues." *Advances in Polymer Technology 32(S1)*: E313-E322.

Saba, N., M. T. Paridah&M. Jawaid (2015). "Mechanical properties of kenaf fibre reinforced polymer composite: A review." *Construction and Building Materials 76*: 87-96.

Sacchetin, P. S. C., R. F. Setti, P. d. T. V. e. Rosa&Â. M. Moraes (2016). "Properties of PLA/PCL particles as vehicles for oral delivery of the androgen hormone 17α-methyltestosterone." *Materials Science and Engineering: C 58*: 870-881.

Saha, A. K., S. Das, D. Bhatta&B. C. Mitra (1999). "Study of Jute Fibre Reinforced Polyester Composites by Dynamic Mechanical Analysis." *Journal of Applied Polymer Science 71*: 1505-1513.

Sain, M., S. H. Park, F. Suhara&S. Law (2004). "Flame retardant and mechanical properties of natural fibre–PP composites containing magnesium hydroxide." *Polymer Degradation and Stability 83(2)*: 363-367.

Sain, M. M., J. Balatinecz&S. Law (2000). "Creep fatigue in engineered wood fibre and plastic compositions." *Journal of Applied Polymer Science 77(2)*: 260-268.

Sajab, M. S., C. H. Chia, S. Zakaria, S. M. Jani, M. K. Ayob, K. L. Chee, P. S. Khiew&W. S. Chiu (2011). "Citric acid modified kenaf core fibres for removal of methylene blue from aqueous solution." *Bioresour Technol 102(15)*: 7237-7243.

Salleh, Z., M.Y.M.Yusop&M.S.Rosdi (2013). "MECHANICAL PROPERTIES OF ACTIVATED CARBON (Abdullah, AlAsady, Ariffin, & Rahman) COIR

FIBRES REINFORCED WITH EPOXY RESIN." *Journal of Mechanical Engineering and Sciences 5*: 631-638.

Salman, S. D., Z. Leman, M. T. H. Sultan, M. R. Ishak&F. Cardona (2016). "Influence of Fibre Content on Mechanical and Morphological Properties of Woven Kenaf Reinforced PVB Film Produced Using a Hot Press Technique." *International Journal of Polymer Science 2016*: 11.

Sapuan, S., M. Harimi&M. Maleque (2003). "Mechanical Properties of Epoxy/Coconut Shell Filler Particle Composites." *Arabian Journal for Science and Engineering 28(2)*: 171-182.

Sapuan S. M., I. H. H., Awang M. and Wiraman R. (2009). "A review of natural fibre reinforced polymer composite research." *Research on Natural Fibre Rinforced Polymer Composites*: 1-12.

Sapuan, S. M.&M. Harimi (2003). "Mechanical properties of epoxy/coconut shell filler particle composites." *The Arabian journal for science and engineering 28(2B)*: 171-181.

Sastra, H. Y., J. P. Siregar, S. M. Sapuan&M. M. Hamdan (2006). "Tensile Properties of Arenga pinnata Fibre-Reinforced Epoxy Composites." *Polymer-Plastics Technology and Engineering 45(1)*: 149-155.

Satyanarayana, K. G., G. G. C. Arizaga&F. Wypych (2009). "Biodegradable composites based on lignocellulosic fibres—An overview." *Progress in Polymer Science 34(9)*: 982-1021.

Shand, M. A. (2006). Magnesia in polymer applications, . *The Chemistry and Technology of Magnesia. Honoken, New Jersey, John Wiley & Sons, Inc*.

Shaw, J. (2000). A Review of Smoke and Potential In-flight Fire Events in 1999*, SAE International*.

Shehata, A. B. (2004). "A new cobalt chelate as flame retardant for polypropylene filled with magnesium hydroxide." *Polymer Degradation and Stability 85(1)*: 577-582.

Shemwell, B. E.&Y. A. Levendis (2000). "Particulates generated from combustion of polymers (plastics)." *Journal of The Air & Waste Management Association, 50(1)*: 94-102.

Shen, H., Y. Wang&K. Mai (2009). "Effect of compatibilizers on thermal stability and mechanical properties of magnesium hydroxide filled polypropylene composites." *Thermochimica Acta 483(1–2)*: 36-40.

Shinoj, S., R. Visvanathan, S. Panigrahi&M. Kochubabu (2011). "Oil palm fibre (OPF) and its composites: A review." *Industrial Crops and Products 33(1)*: 7-22.

Shukor, F., A. Hassan, M. Saiful Islam, M. Mokhtar&M. Hasan (2014). "Effect of ammonium polyphosphate on flame retardancy, thermal stability and mechanical properties of alkali treated kenaf fibre filled PLA biocomposites." *Materials & Design (1980-2015) 54*: 425-429.

Small, A. C., M. Rogers, L. Sterner, T. Amos&A. Johnson (2007). "A Novel Non-Halogenated Flame Retardant for Composite Materials." *Composites Research Journal 1(2)*.

Smith, W. F.&J. Hashemi (2006). Composite materials. *Foundations of Materials Science and Engineering. New York, McGraw-Hill.* ***5***.

Society, R. A. (2013). "Smoke, fire and fumes in transport aircraft: Past history, current risk and recommended mitigations." 2015, from http://www.inflightwarningsystems.com/AAfiles/articles/aerosociety2013.pdf.

Srebrenkoska, V., D. Dimeski, G. Bogoeva-Gaceva, A. Grozdanov&A. Buzarovska (2006). CARACTERIZATION OF KENAF / PLA COMPOSITES

`

International Conference of the Chemical Societies of the South-East European Countries

Sreekumar, P. A., P. Gopalakrishnan, N. Leblanc&J. M. Saiter (2010). "Effect of glycerol and short sisal fibres on the viscoelastic behavior of wheat flour based thermoplastic." *Composites Part A: Applied Science and Manufacturing 41(8)*: 991-996.

Stark, N. M., R. H. White, S. A. Mueller&T. A. Osswald (2010). "Evaluation of various fire retardants for use in wood flourepolyethylene composites." *Polymer Degradation and Stability 95*: 1903-1910.

Subasinghe, A.&D. Bhattacharyya (2014). "Performance of different intumescent ammonium polyphosphate flame retardants in PP/kenaf fibre composites." *Composites Part A: Applied Science and Manufacturing 65*: 91-99.

Suharty, N. S., I. P. Almanar, Sudirman, K. Dihardjo&N. Astasari (2012). "Flammability, Biodegradability and Mechanical Properties of Bio-Composites Waste Polypropylene/Kenaf Fibre Containing Nano CaCO3 with Diammonium Phosphate." *Procedia Chemistry 4*: 282-287.

Suharty, N. S.&M. Firdaus (2006). Synthesis of degradable bio-composites polystyrene recycle modified in reactively process using natural fibre filler. *Proceeding of 12th Asian Chemical Congress,, Kuala Lumpur, Malaysia*.

Summerscales, J., N. P. J. Dissanayake, A. S. Virk&W. Hall (2010). "A review of bast fibres and their composites. Part 1 – Fibres as reinforcements." *Composites Part A: Applied Science and Manufacturing 41(10)*: 1329-1335.

Suppakarn, N.&K. Jarukumjorn (2009). "Mechanical properties and flammability of sisal/PP composites: Effect of flame retardant type and content." *Composites Part B: Engineering 40(7)*: 613-618.

Tan, B. K., Y. C. Ching, S. N. Gan, S. Ramesh&M. R. Rahman (2014). "Water absorption properties of kenaf fibre–poly(vinyl alcohol) composites." *Materials Research Innovations 18(sup6)*: S6-144-S146-146.

Tang, C. Y.&J. Z. Liang (2003). "A study of the melt flow behaviour of ABS/CaCO3 composites." *Journal of Materials Processing Technology 138(1–3)*: 408-410.

Tawakkal, I. S. M. A., M. J. Cran&S. W. Bigger (2014). "Effect of kenaf fibre loading and thymol concentration on the mechanical and thermal properties of PLA/kenaf/thymol composites." *Industrial Crops and Products 61*: 74-83.

Tesoro, G. C. (1978). "Chemical modification of polymers with flame-retardant compounds." *Journal of Polymer Science: Macromolecular Reviews 13(1)*: 283-353.

Tewarson, A. (1994). "Flammability parameters of materials: Ignition, combustion and fire propagation." *Journal of Fire Sciences 12*: 329-355.

Thakur, V. K.&M. K. Thakur (2014). "Processing and characterization of natural cellulose fibres/thermoset polymer composites." *Carbohydrate Polymers 109*: 102-117.

Thomas, S., D. Durand, C. Chassenieux&P. Jyotishkumar (2013). Handbook of Biopolymer-Based Materials: From Blends and Composites to Gels and Complex Networks*, Wiley.*

Thomas, S.&L. A. Pothan (2009). Natural Fibre Reinforced Polymer Composites: From Macro to Nanoscale*, Éd. des Archives Contemporaines.*

Tian, H., Y. X. Zhang, L. Ye&C. Yang (2015). "Mechanical behaviours of green hybrid fibre-reinforced cementitious composites." *Construction and Building Materials 95*: 152-163.

Tolf, G. r.&P. Clarin (1984). "Comparison between Flexural and Tensile Modulus of

Fibre Composites." *Fibre Science and Technology 21* 319-326.

Transportation, U. S. D. o. (2000). Chapter 3: Safety, Dureau of Transportation Statistics *The Changing Face of Transportation. Washington, DC.*

Tsuji, H.&K. Ikarashi (2004). "In vitro hydrolysis of poly(L-lactide) crystalline residues as extended-chain crystallites, Part I: long-term hydrolysis in phosphate buffered solution at 37 degrees C." *Biomaterials 25*: 5449-5455.

Tsuji, H.&K. Ikarashi (2004). " In vitro hydrolysis of poly(l-lactide) crystalline residues as extended-chain crystallites: II. Effects of hydrolysis temperature." *Biomacromolecules 5(3)*: 1021-1028.

Venkateshwaran, N.&A. Elayaperumal (2010). "Banana Fibre Reinforced Polymer Composites - A Review." *Journal of Reinforced Plastics and Composites 29(15)*: 2387-2396.

Vijaya Ramnath, B., S. Junaid Kokan, R. Niranjan Raja, R. Sathyanarayanan, C. Elanchezhian, A. Rajendra Prasad&V. M. Manickavasagam (2013). "Evaluation of mechanical properties of abaca–jute–glass fibre reinforced epoxy composite." *Materials & Design 51*: 357-366.

Vink, E. T. H., K. R. Rábago, D. A. Glassner&P. R. Gruber (2003). "Applications of life cycle assessment to NatureWorks™ polylactide (PLA) production." *Polymer Degradation and Stability 80(3)*: 403-419.

Wang, W., A. Lowe, S. Davey, N. Akhavan Zanjani&S. Kalyanasundaram (2015). "Establishing a new Forming Limit Curve for a flax fibre reinforced polypropylene composite through stretch forming experiments." *Composites Part A: Applied Science and Manufacturing 77*: 114-123.

Warringtonfire, E. (2010). "Fire testing and fire consultancy." 2015, from https://www.exova.com/sectors/fire/.

Witzke D. R. (1997). Introduction to properties, engineering, and prospects of polylactide polymers. Department of Chemical Engineering*, Michigan State Univeristy, East Lansing, MI.*

Xiao L., W. B., Yang G. and Gauthier M. (2012). "Poly(lactic acid)-based biomaterials: synthesis, modification and applications, ." *Biomedical Science, Engineering and Techonology, Intech, Croatia*.

Xu, J., Y. Jiao, B. Zhang, H. Qu&G. Yang (2006). "Tin dioxide coated calcium carbonate as flame retardant for semirigid poly(vinyl chloride)." *Journal of Applied Polymer Science 101(1)*: 731-738.

Yahaya, R., S. M. Sapuan, M. Jawaid, Z. Leman&E. S. Zainudin (2015). "Effect of layering sequence and chemical treatment on the mechanical properties of woven kenaf–aramid hybrid laminated composites." *Materials & Design 67*: 173-179.

Yallew, T. B., P. Kumar&I. Singh (2014). "Sliding Wear Properties of Jute Fabric Reinforced Polypropylene Composites." *Procedia Engineering 97*: 402-411.

Yan, Z. L., H. Wang, K. T. Lau, S. Pather, J. C. Zhang, G. Lin&Y. Ding (2013). "Reinforcement of polypropylene with hemp fibres." *Composites Part B: Engineering 46*: 221-226.

Yang, H.-S., H.-J. Kim, J. Son, H.-J. Park, B.-J. Lee&T.-S. Hwang (2004). "Rice-husk flour filled polypropylene composites; mechanical and morphological study." *Composite Structures 63(3–4)*: 305-312.

Yang, J., J. Z. Liang&C. Y. Tang (2009). "Studies on melt flow properties during capillary extrusion of PP/Al(OH)3/Mg(OH)2 flame retardant composites." *Polymer Testing 28(8)*: 907-911.

Yang, Y., T. Ota, T. Morii&H. Hamada (2011). "Mechanical property and hydrothermal aging of injection molded jute/polypropylene composites." *Journal of Materials Science 46(8)*: 2678-2684.

Yoo, D. K., D. Kim&D. S. Lee (2006) "Synthesis of lactide from oligomeric PLA: Effects of temperature, pressure, and catalyst." *Macromolecular Research 14(5)*: 510-516.

Yousfi, M., S. Alix, M. Lebeau, J. Soulestin, M.-F. Lacrampe&P. Krawczak (2014). "Evaluation of rheological properties of non-Newtonian fluids in micro rheology compounder: Experimental procedures for a reliable polymer melt viscosity measurement." *Polymer Testing 40*: 207-217.

Yousif, B. F., A. Shalwan, C. W. Chin&K. C. Ming (2012). "Flexural properties of treated and untreated kenaf/epoxy composites." *Materials & Design 40*: 378-385.

Zainudin, E. S., S. M. Sapuan, K. Abdan&M. T. M. Mohamad (2009). "Thermal degradation of banana pseudo-stem filled unplasticized polyvinyl chloride (UPVC) composites." *Materials & Design 30(3)*: 557-562.

Zampaloni, M., F. Pourboghrat, S. A. Yankovich, B. N. Rodgers, J. Moore, L. T. Drzal, A. K. Mohanty&M. Misra (2007). "Kenaf natural fibre reinforced polypropylene composites: A discussion on manufacturing problems and solutions." *Composites Part A: Applied Science and Manufacturing 38(6)*: 1569-1580.

Zebarjad, S. M., S. A. Saijadi, M. Tahani&A. Lazzeri (2006). "A study on the thermal behaviour of HDPE/CaCO3 nanocomposites." *Journal of Achievements in Materials and Manufacturing Engineering 7(1-2)*: 173-176.

Zhang, S.&A. R. Horrocks (2003). "A review of flame retardant polypropylene fibres." *Progress in Polymer Science 28(11)*: 1517-1538.

Zhang, Z. X., J. Zhang, B.-X. Lu, Z. X. Xin, C. K. Kang&J. K. Kim (2012). "Effect of flame retardants on mechanical properties, flammability and foamability of PP/wood–fibre composites." *Composites Part B: Engineering 43(2)*: 150-158.

Zheng, C., Y. Xu, T. Kawai&S. Kuroda (2013). "Effect of polymeric coupling agent on mechanical properties of kenaf fibre/polystyrene composites." *Appl. Mech. Mater 268* 127- 133.

`

APPENDIXES

APPENDIX A

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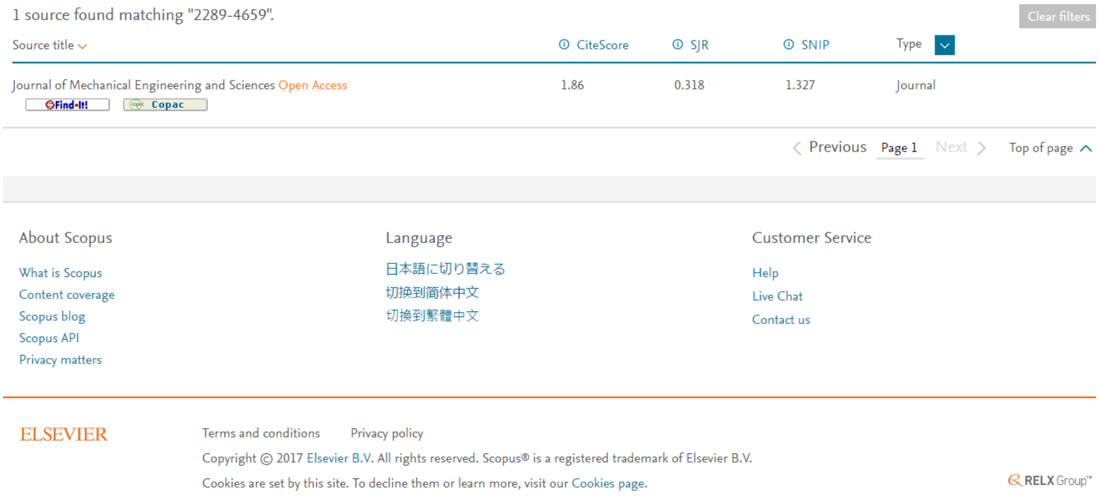

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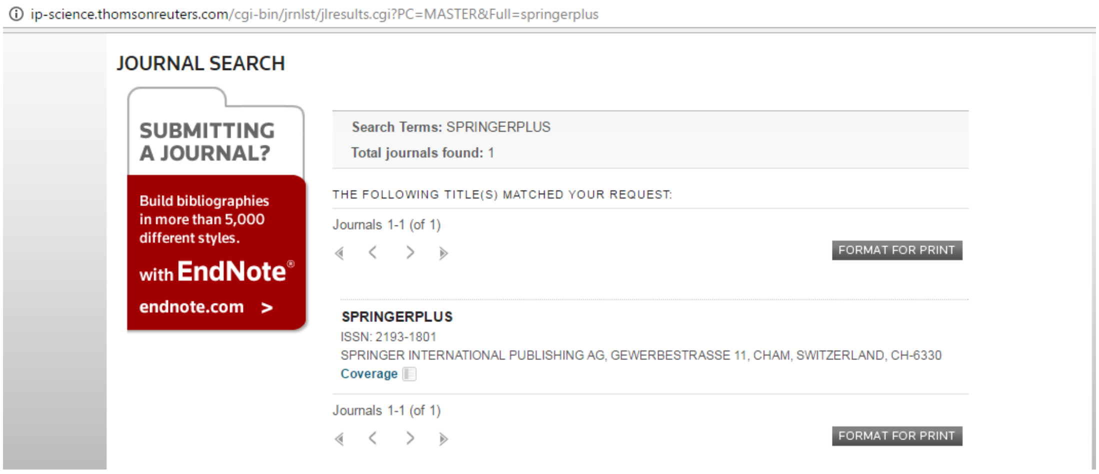

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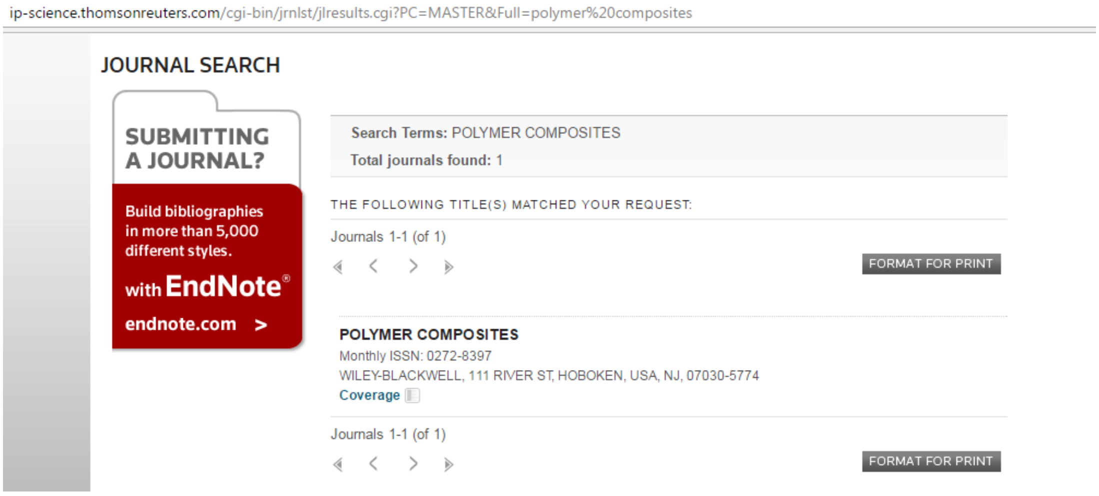

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LIST OF PUBLICATION

1. Khor, L.S., Leman, Z., Lee, C.H. Interfacial debonding force and shear strength of sugar palm (arenga pinnata) fiber reinforced composites by pull-out test (2013) Advanced Materials Research, 634-638 (1), pp. 1931-1936.

2. C. H. Lee, S. M. Sapuan, M. R. Hassan, A Review of the Flammability Factors of Kenaf and Allied Fibre Reinforced Polymer Composites, Advances in Materials Science and Engineering, Volume 2014 (2014)

3. C. H. Lee, S. M. Sapuan, M. R., Thermal Analysis of Kenaf Fiber Reinforced Floreon Biocomposites with Magnesium Hydroxide Flame Retardant Filler, Polymer Composite

4. C. H. Lee, S. M. Sapuan, J. H. Lee, M. R. Hassan, Melt Volume Flow Rate and Melt Flow Rate of Kenaf Fibre Reinforced Floreon/ Magnesium Hydroxide Biocomposites, Springerplus, 2016,5(1), 1680

5. C. H. Lee, S. M. Sapuan, J. H. Lee, M. R. Hassan, Mechanical properties of kenaf fibre reinforced floreon biocomposites with magnesium hydroxide filler, Journal of Mechanical Engineering and Sciences, 10(3), 2016, pp 2234-224.

6. C. H. Lee, S. M. Sapuan, J. H. Lee, M. R. Hassan, Mechanical and Thermal Properties of Kenaf Fiber Reinforced Polypropylene/Magnesium Hydroxide Composites, Journal of Engineered Fibers and Fabrics, 12(2), 2017, pp 50-58.