Pretreatment Of Binder With Ionic Liquid For The Fabrication Of Eco-Friendly Composite

By

Chiah Yoke Yi

15106

Dissertation submitted in partial fulfillment of
the requirements for the
Degree of Study (Hons)
Chemical Engineering
FYP II Semester II Year 4

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Chemical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfillment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(CHEMICAL)

Approved by,

(Dr. Muhammad Moniruzzaman)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2014

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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(CHIAH YOKE YI)

ACKNOWLEDGEMENT

Foremost, I feel very thankful because the Final Year Project (FYP) is finally completed smoothly and successfully. Definitely, the successful completion of this project is the contributions from many individuals and I feel very grateful for their support and guidance. In this project, I was able to expose to the different types of ionic liquids and their unique properties, the pretreatment of oil palm biomass with ionic liquids, thermal-compression molding technique as well as the different types of biocomposite board properties testings which includes FT-IR, TGA and DSC. Besides, I also learned about paper research as well as the project planning and scheduling. In short, I have gained a lot of new experience and knowledge from this project.

Firstly, I would like to give my deepest appreciation and gratefulness to my FYP supervisor, Dr. Muhammad Moniruzzaman. Being my supervisor since my internship, he is very helpful and willing to guide me patiently on all the problems that I am facing with. I feel very grateful on his trust on me in completing the project.

Furthermore, I would like to express my thankfulness to Mr. Hamayoun Mahmood who is a post-graduate student of my supervisor. Despite of being busy of his own duties, he had guided me patiently throughout my FYP in delivering the project. He is also willing to share his experience and knowledge in his studies with me.

In addition, I would like to give special thanks to all the laboratory technicians who had assisted me in conducting the experiments and testings. Mr. Kamarul and Mr. Danial from Block 17 laboratory had guided me on operating the thermal-compression molding machine; laboratory technicians Centralized Analytical Laboratory (CAL) had helped to conduct the FT-IR testing and post-graduate students from block 4 had guided me on the TGA and DSC testings.

Last but not least, I would like to give my special appreciation to Universiti Teknologi PETRONAS (UTP) for providing opportunity for the students to deliver a final year project which expose them to work independently while providing conducive environment, equipment and instruments to complete the projects.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLE

ABSTRACT

Synthetic composites have many applications and its rapid gloom started since 1960s. However, synthetic composites are produced from the non-renewable resource which is the petroleum. Besides, the manufacturing process of synthetic composites has caused a lot of environmental pollution and the non-biodegradable of synthetic composites has lead to disposal problem. Thus, research starts to produce bio-degradable, renewable, and clean manufacturing process biocomposites. The result from the previous studies showed that generally, the biocomposite films possess good mechanical properties and thermal stability.

In this study, a biocomposite boards will be prepared by utilizing the oil palm frond (OPF) and palm kernel shell (PKS) mixed with the thermo-plasticized starch (TPS) which is prepared by treating with different types of ionic liquids (1-ethyl-3-methylimidazolium diethyl phosphate, 1-butyl-3-methylimidazolium chloride, 1-ethyl-3-methylimidazolium acetate, 1-butyl-3-methylimidazolium trifluoromethane sulfonate). The effect of the processing parameters on the properties of the biocomposite boards are investigated. The parameters to be investigated include the temperature, pressure and the time of thermal compression to produce the biocomposite boards. Besides, the influence of different types of ionic liquid on the TPS performance as an external binder will also be investigated. The properties to be included in the study focused on the internal bonding and the thermal stability of the biocomposite boards. The internal bonding of the biocomposite boards were examined using the Fourier Transform Infrared Spectroscopy (FTIR) whereas the thermal stability was examined using Differential Scanning Calorimetry (DSC), Thermo Gravimetry Analysis (TGA). The FT-IR result revealed that there the presence of IL (except BmimTFMS) help to induce the inter-hydrogen bond formation. The TGA result showed that the biocomposite boards are thermally stable until very high temperature (>250^{o}C). The successful preparation of OPF-PKS biocomposite boards with TPS will become the insight of green chemistry which eliminates the use or the generation of non-environmental friendly substances.

CHAPTER 1

INTRODUCTION

1. 1 BACKGROUND OF STUDY

1.1.1 Synthetic Composites

Synthetic composites are the composites derived from non-naturally occurring polymers such as polyethylene, polystyrene, epoxies, polyvinyl chloride (PVC), and so on which are in turn obtained from the non-renewable source of petroleum. The global production of synthetic polymers is in the increasing trend. This is to respond to the increasing demand in the synthetic polymer products. In fact, it has even become a part of human daily lives that cannot be lack of. No doubt, synthetic polymers do play many important roles in our daily lives such as the plastic bags, constructions, automotive, furniture, clothes and spare parts of many different equipment and even military and aircraft. The wide applications of synthetic composites is due to its good mechanical strength, corrosive resistant, and high chemical stability. Despite of all of these important roles, the productions of synthetic polymers have created serious problems to the environment (Anil, N. N., & Chabba, S., 2003).

1.1.2 Controversies Against Synthetic Composites

There are few reasons to support this argument. Firstly, the synthetic polymers are produced from petroleum. From the drilling of petroleum until the downstream processing of it, the whole process has contributed a lot to the environmental pollution. Apart from that, the production of synthetic polymers and the improper disposal of the synthetic polymer products which are non-biodegradable have further magnify the problem. Next, the synthetic composites are very difficult to be recycled and reuse. This is because the synthetic composites are composed of two or more types of polymers, which makes them to be difficult to be recycled easily. The only way to manage the synthetic composite wastes are to decomposed using incinerator or landfill. Both of these methods are causing environmental pollution (La Mantia, F. P., & Morreale, M., 2011).

1.1.3 Development of Biocomposites

Global awareness about the environmental protection, stricter laws and the depletion of petroleum have caused many parties become more concern about the drawbacks of synthetic composites and try to determine the alternatives which are eco-friendly, biodegradable, and sustainable to substitute the synthetic polymers. (Anil, N. N., & Chabba, S., 2003). Previously, efforts have been used to incorporate biopolymers as biodegradable fillers or reinforcement agent in synthetic composites. However, the synthetic biodegradable composites are still relies on the petroleum resource and there is the compatibility problems between the natural polymers and synthetic polymers. This is followed by the development of blend of natural polymer together with its derivatives such as cellulose acetate, starch acetate and so on. This method solved the compatibility problems of the polymers but the drawback is that the derivatives are expensive (Wu, R. L., et al., 2009).

Thus, attention has been put on producing biocomposites from solely natural polymers. Biocomposite is the composite derived from naturally-occurring polymers such as starch, cellulose, lignin and so on, which can be obtained from the wood, cotton, wool, silk and so on. It has been reported that cellulose functions to provide mechanical strength on the biocomposite. On the other hand, starch may contribute to the film flexibility and lignin can act as a natural binding agent of cellulose (Wu, R. L., et al., 2009). However, the processing of most of the biopolymers are very difficult due to the strong hydrogen bonding, high degree of polymerization and crystallinity of the biopolymers. Even though there are existing solvent to dissolve the biopolymers, the solvent are highly volatile, toxic, instable, expensive, difficult to be regenerated and require high energy consumption.

1.1.4 Ionic Liquid (IL)

Ionic liquid (IL) provides good insight as a solvent for biopolymers. This is because it possess unique properties which include extreme low volatility, high thermal and chemical stability, biodegradable and the ability to be regenerated. IL is generally salts with melting point of less than 100°C. The properties such as viscosity, melting point and hydrophobicity can be altered by manipulating the cation or anion. Thus, IL is a green solvent for biopolymers (Hameed, N., et al, 2010).

1.1.5 Sources Of Biopolymers

There are many source of biopolymers as discussed above. Previous research were done on wool, cotton, silk, cellulose, starch, chitosan and lignin. Apart from this, oil palm frond (OPF) and palm kernel shell (PKS) is also a source of cellulose, hemicellulose, starch and lignin. OPF and PKS is the leftover of the oil palm fruit extract. OPF is widely being used as soil nutrient, formulated feed for ruminants and fuel for boilers and power plants. On the other hand, PKS can be used to make charcoal, activated carbon, light weight aggregate for concrete, as well as an alternative to substitute non-renewable fuel since it has high calorific value and low ash and sulfur content in the combustion products. Other oil palm trees leftover includes the oil palm empty fruit bunch (OPEFB). It has been used for many applications in different fields which includes the production of wood-based products, paper and pulp productions, electricity generation, mulch, horticulture, organic fertilizer and so on.

1.2 PROBLEM STATEMENT

The production and consumption of synthetic polymer products has lead to serious environmental pollution. Effort has been put on finding out the alternatives to substitute or reduce the dependence on synthetic polymers. The existing wood based composite utilize formaldehyde resin as the binder which acts as an agent to bind the component polymers that make up the composite. However, formaldehyde is carcinogenic and bad to human health.

One potential source of natural polymers to make biocomposite is OPF, OPEFB and PKS. The oil palm raw biomass in Malaysia is not fully utilized and this causes there are still a vast amount of the raw materials being disposed as waste into the environment. This has caused waste management problem in the oil palm sector. In 2009, 20.41 million tons of OPEFB, 5.32 million tons of PKS and 6.89 million tons of OPF are generated (Oil Palm Products, n. d.). Some of these raw materials are used to generate electricity, to produce fertilizer, to manufacture papers and so on. The excess raw materials will be disposed to the environment and it will decompose which becomes a source of nutrients to the soil, making the surrounding plants and trees to grow more fertile. Despite of this, the disposal these raw materials is not a good means because there is economic potential to be uncovered, and one of the possibilities is to produce biocomposite.

Research was done previously to prepare biocomposite films using oil palm biomasses without the pretreatment with ionic liquid. The biocomposite films produced have achieved certain standard of strength. Yet, the data on the effect of different parameters on the properties of OPF, PKS and OPEFB biocomposite is still insufficient until now and the effect of ionic liquid pretreatment on external binder to the mechanical strength and thermal stability of the boards are yet to be discovered.

1.3 OBJECTIVES

The purpose of this final year project is to achieve few objectives as below:

i) To study the effect of different IL pretreatment on the preparation of thermo-plasticized starch (TPS).

ii) To synthesize eco-friendly biocomposite boards using OPF and PKS with the addition of TPS by thermal compression molding technique. This project open the insight to produce composite which are biodegradable, the eco-friendly production process and reduction in the dependence of synthetic polymers from the non-renewable petroleum.

iii) Use different equipment which include FTIR, TGA, DSC and so on to analyze the properties of the biocomposite boards. The correct procedure is to be followed to prevent any damage to the equipment and also minimize the error in the data. The function, procedure and method of operating different equipment need to be understood prior to carrying out the experiment.

iv) To study the effect of different parameters on the physical and thermal properties of the biocomposite boards produced using OPF and PKS. The properties of the biocomposite boards produced under different parameters will then be compared. The data obtained can be used as a reference in the future to manufacture the biocomposite with the improved physical, mechanical and thermal properties by adjusting the parameters to the optimum level.

1.4 SCOPE OF STUDY

Biocomposites have wide scope and potentials to be applied in many different fields. Firstly, it can be used in construction field whereby it can be used as reinforcement material in structural concrete, roofting and cementing. Furthermore, biocomposites can be used in electronic material applications whereby it is used to make low dielectric constant material which includes the microchips and circuit boards. In addition, biocomposites also have the potential to benefit the automotive industries in the manufacturing of car disk brakes, dashboards, door panels and mat. The biocomposite applications in military and aerospace still require more research because very precise and high quality of the mechanical and thermal properties are required in these applications.

In this project, biocomposite boards will be prepared by using raw OPF and PKS and mix with TPS pretreated with IL. The mixture will then undergo thermal compression to compress the biomass powder into biocomposite hardboards during heating. The biocomposites properties were studied using the FTIR, DSC and TGA. FTIR is used to study the bonding between the molecules in the biocomposites. Both DSC and TGA is used to study the thermal stability of the films where DSC measures the heat absorbed or released with increasing temperature and TGA measures the weight percent of sample with temperature.

If there is time allowance, the water absorption, thickness swelling and mechanical properties will be analyzed which compressive strength, tensile strength, yield strength and modulus. Compressive strength is the maximum compression force before failure of the material. Tensile strength is the maximum tensile stress before failure of the material. Yield strength is the stress where the material starts to yield. Modulus is the ratio of stress to strain.

The parameters to be considered in this project include the types of ionic liquid used as a solvent during TPS preparation, the temperature and pressure of the thermal compression molding.

CHAPTER 2

LITERATURE REVIEW

Some literatures are studied and understood to get some reference for this project. The first part of the summary consists of the literatures that discusses on the biocomposite preparation with IL as solvent to the biomass. In order to increase the binding strength of the component polymers, TPS which acts as a binder in the biomass is included. Several literatures related to TPS were also studied. From the previous research, it is found out that ionic liquid is act as a good plasticizer for starch. The significance and limitation of each literatures are summarized in tabular form as follow.

TABLE 1: Summary of the literature review.

Literature	Significance/Findings	Limitations/Gap
Wu, R. L., Wang, X. L., Li, H. Z., & Wang, Y. Z. (2008). Green Composite Films Prepared From Cellulose, Starch And Lignin In Room-Temperature Ionic Liquid. *Bioresource Technology,* 100, 2009, 2569–2574.	-Biocomposite films prepared from cellulose, starch and lignin with 1-allyl-3-methylimidazolium chloride (AmimCl) used as solvent. -Cellulose and lignin give significant effect on the mechanical properties of the films whereas starch gives the films flexibility. - Biocomposite films of different composition of components were prepared and investigated. -The films possess good thermal stability and gas permeability.	-The effect of different parameters (types of biopolymer , temperature, pressure, types of IL, source of the biopolymer) during the preparation of biocomposite films not included. - The effect of presence of external binder to the biocomposite properties not included.

	Films have good mechanical properties due to the mutual supplement of the components. Proved that IL is an environmental friendly solvent for biopolymer.	
Abdulkhani, A., Marvast, E. H., Ashori, A., Hamzeh, Y., & Karimi, A. N. (2013). Preparation Of Cellulose/Polyvinyl Alcohol Biocomposite Films Using1-N-Butyl-3-Methylimidazolium Chloride. *International Journal of Biological Macromolecule,* 62, 2013, 379-386.	- Biocomposite films were prepared using cellulose and polyvinyl alcohol (PVA) with 1-n-butyl-3-methylimidazolium chloride (BmimCl) as a solvent. - Biocomposite films of different composition of components were prepared and investigated. - Biocomposite has lower crystallinity than raw cellulose. - The thickness swelling, water uptake, water vapour permeability and strain at break of the films were improved compared to the raw cellulose. - The mechanical strength and Young's modulus of the films were decreased. - Cellulose contribute to the improved thermal stability of the biocomposite films.	- Only cellulose is investigated. - The effect of different parameters (types of biopolymer , temperature, pressure, types of IL, source of the biopolymer) during the preparation of biocomposite films not included. - The effect of presence of external binder to the biocomposite properties not included. - PVA is synthetic polymer.
Silva, R. D., Wang, X., & Byrne, N. (2013). Tri-Component Bio-Composite	- Biocomposite films prepared from cotton, silk and wool using 1-allyl-3-methylimidazolium (AmimCl) as the solvent. - Investigated the effect of	- The component and composition of the cotton, silk and wool not identified. - The effect of different

Materials Prepared Using An Eco-Friendly Processing Route.	different composition of the blends and the types of coagulating agents on the physical and thermal properties of the biocomposite films. - The wool and silk boost the thermal degradation temperature of the biocomposite films which can be explained by the intra molecular hydrogen bonds in the biocomposite. - The coagulating agent does affect the properties of the films and water to be the best coagulating solvent.	parameters (types of biopolymer ,temperature, pressure, types of IL, source of the biopolymer) during the preparation of biocomposite films not included. - The effect of presence of external binder to the biocomposite properties not included.
Hameed, N., & Guo, Q. (2010). Blend films of natural wool and cellulose prepared from an ionic liquid.	- Biocomposite films were prepared using natural wool and cellulose and 1-butyl-3-methylimidazolium chloride (BmimCl) as solvent. - Investigated the effect of different wool/cellulose composition on the biocomposite films properties. - The blend films possessed improved thermal stability and elongations due to the hydrogen bonding formation between the two species. - Cellulose content accounts for the improvement of film thermal stability and tensile strength. - Opened an insight on the	- The effect of different parameters (types of biopolymer, temperature, pressure, types of IL, source of the biopolymer) during the preparation of biocomposite films not included. - The effect of presence of external binder to the biocomposite properties not included.

	possibility of producing renewable biocomposite with improved properties.	
Stefanescu, C., Daly, W. H., & Negulescu, I. I. (2011). Biocomposite Films Prepared From Ionic Liquid Solutions Of Chitosan And Cellulose.	- Biocomposite films were prepared from cellulose and chitosan with 1-butyl-3-methylimidazolium acetate (BmimAc) as solvent. - Investigated the effect of different composition of cellulose and chitosan on the biocomposite films properties. - Proved that cellulose and chitosan are miscible in the presence of IL.	- The effect of different parameters (types of biopolymer, temperature, pressure, types of IL, source of the biopolymer) during the preparation of biocomposite films not included. - The effect of presence of external binder to the biocomposite properties not included. - No mechanical test is conducted.
Takegawa, A., Murakami, M., Kaneko, Y., & Kadokawa, J. (2009). Preparation Of Chitin/Cellulose Composite Gels And Films With Ionic Liquids.	- Biocomposite films were prepared from cellulose and chitin with AmimBr and BmimCl as solvents. - The effect of different composition of cellulose, chitin, AmimBr and BmimCl on the biocomposite films properties were investigated. - The crytallinity of the biopolymers were destroyed which proved the miscibility between two biopolymers under the presence of ionic liquid. - Gels became more brittle when the chitin composition is	- The effect of different parameters (types of biopolymer, temperature, pressure, types of IL, source of the biopolymer) during the preparation of biocomposite films not included. - The effect of presence of external binder to the biocomposite properties not included.

	increased. - Films became more elastic when the chitin composition is decreased.	
Zhao, D., Li, H., Zhang, J., Fu, L., Liu, M., Fu, J., et al. (2011). Dissolution Of Cellulose In Phosphate-Based Ionic Liquids.	- Studied the effect of dissolution temperature on the dissolution time and the degree of polymerization of cellulose on the solubility of cellulose in IL (phosphate-based). - Dissolution time was greatly reduced at higher dissolution temperature. - Dissolution should be less than 100°C to maintain degree of polymerisation stability of the regenerated cellulose. - Regenerated cellulose improved thermal stability.	- Research only focus on cellulose. - The effect of presence of external binder to the biocomposite properties not included.
Xiao, W., Chen, Q., Wu, Y., Wu, T., & Dai, L. (2010). Dissolution And Blending Of Chitosan Using 1,3-dimethylimidazolium Chloride And 1-H-3-Methylimidazolium Chloride Binary Ionic Liquid Solvent.	- Studied the solubility of chitosan and cellulose in different ionic liquids and the properties of the films produced. - Dissolution power of ionic liquid decreases from 1,3-dimethylimidazolium chloride(DmimCl) to AmimCl, and finally BmimCl. - Ionic liquid with lower viscosity will ease the regeneration of cellulose. - Good miscibility of cellulose and chitosan was obtained and the	- The effect of presence of external binder to the biocomposite properties not included. - The research only study chitosan and cellulose.

	regenerated chitosan have better thermal stability than raw chitosan. - Blended films were more brittle when more chitosan is present.	
Zhang, J., Wang, Y., Zhang, L., Zhang, R., Liu, G., & Cheng, G. (2013). Understanding Changes In Cellulose Crystalline Structure Of Lignocellulosic Biomass During Ionic Liquid Pretreatment By XRD.	- Studied the interactions of cellulose in the presence of ionic liquid (1-butyl-3-methylimidazolium acetate). - The dissolution of cellulose in ionic liquid occur through the progressive swell of the biomass. - The swollen part of the biomass was then transformed into a disordered structure which remain even after treated with anti-solvent (water). This causes the fall of biomass crystallinity index (CrI).	- Research only focus on cellulose. - No thermal and mechanical strength test included.
Patachia, S., Croitoru, C., & Rusu, V. (2013). Obtaining Of Ecological Composites From Wood Waste Using Ionic Liquids.	- Prepared biocomposites using wood waste with BmimCl as solvent. - Mechanical strength and moisture absorption were studied. - Pretreated biocomposites possessed improved compression strength and dimensional stability under different moisture environment compared to the raw biocomposites.	- No thermal stability test included.

Feng, L., & Chen, Z. I. (2008). Research Progress On Dissolution And Functional Modification Of Cellulose In Ionic Liquids.	- Summarized the previous researches on the dissolution and functional modification of cellulose using ionic liquid. - Solubility of cellulose is negative related with the solvent carbon chain and the cellulose degree of polymerization. - The IL can be designed to have specific function by adjusting the cation and anion.	- Only focus on the dissolution of cellulose in IL.
Leroy, E., Jacquet, P., Coativy, G., Reguerre, A. L., & Lourdin, D. (2012). Compatibilization Of Starch-Zein Melt Processed Blends By An Ionic Liquid Used As Plasticizer.	- Studied the effect of plasticizers, 1-butyl-3-methylimidazolium chloride (BmimCl) and glycerol on the properties of thermoplastic plasticized starch and zein. - Both glycerol and ionic liquid are able to plasticize the starch and zein. - Both plasticizers have significant effect on the melt viscosity of starch but not for zein. - BmimCl is a better plasticizer compared to glycerol (The starch is compatible with zein in the starch/zein blend in the presence of BmimCl but the mixture is incompatible in the presence of glycerol).	- The effect of different IL and the origin of starch on the plasticizing effect not included.
Sankri, A., Arhaliass, A., Dez, I., Gaumont, A. C.,	- Studied the plasticizing strength of BmimCl and glycerol towards starch.	- The effect of different IL and the origin of starch on the plasticizing effect not

Grohens, Y., Lourdin, D., et al. (2010). Thermoplastic Starch Plasticized By An Ionic Liquid.	- BmimCl is a better plasticizer of starch. - Thermo plasticized starch (TPS) has lower water uptake, higher elongation at break, lower glass transition temperature, lower Young's modulus. - Low value of Young's modulus is due to the reduction of hydrogen bond between the starch chain in the presence of ionic liquid.	included.
Selamat, M. E., Sulaiman, O., Hashim, R., Hiziroglu, S., Wan Nadhari, W. N. A., Sulaiman, N. S., et al. (2014). Measurement Of Some Particleboard Properties Bonded With Modified Carboxymethyl Starch Of Oil Palm Starch.	- Produced and measured the mechanical and physical properties of particleboard using rubber wood and different external binder (native starch, carboxylmethyl starch and carboxylmethyl starch mixed with 2% urea formaldehyde). - Carboxylmethyl starch produces particleboard with better mechanical and physical properties compared to starch alone. - The properties of particle boards is improved by meeting the requirements of Type 8 particleboard in Japanese Standard in the presence of urea formaldehyde.	- Urea formaldehyde is not a natural occurring adhesive. - Carboxymethyl starch is produced by chemically modifying the starch with phosphoryl chloride ($POCl_3$) which means the binder is not purely natural-occurring binder.
Amini, M. H. M., Hashim, R.,	- Prepared and studied the particleboard made from rubber	- Modified starch is not purely natural occurring

Hiziroglu, S., Sulaiman, N. S., Sulaiman, O. (2013). Properties Of Particleboard Made From Rubberwood Using Modified Starch As Binder.	wood and modified starch (native starch mixed with glutardialdehyde) as the binder. - Result satisfies the Japanese Industrial Standard (JAS) with the water absorptivity needed to be further improved with water repellent materials.	binder.
Wilpiszewska, K., & Spychaj, T. (2011). Ionic Liquids: Media For Starch Dissolution, Plasticization And Modification.	- Studied the different researches regarding the function of ionic liquid as a media for starch dissolution, modification and plasticization. - Ionic liquid can act as solvent or plasticizer for pure starch. - Starch modification by ionic liquid includes acid-catalyzed hydrolysis, esterification, graft copolymerization and so on.	- The effect of anion and cation types on alkyl substituents of IL not included. - The effect of the origin of starch on the behavior with IL is not included.

From the above table, it can be concluded that IL is a good solvent to naturally occurring biopolymers and binder (starch) in the preparation of TPS. The research on the preparation of biocomposite using oil palm biomasses are limited and the different preparation parameters on the thermal and mechanical properties of the biocomposite hardboard require more further research.

CHAPTER 3

METHODOLOGY

3.1 METHODOLOGY

The project research is divided into two parts. The first part involved the preparation of biocomposite hardboard through the IL pretreatment of oil palm biomass. The second part involved the adding of binder pretreated with IL onto the raw biomass instead of the direct pretreatment of biomass with IL. This is because the result of the first part of the research is not satisfying. The research methodology will start with sample preparation. The methodology is discussed in details as follows:

3.1.1 Methodology (Initial Stage Of The Project)

i) Sample preparation

OPEFB will be collected from local oil palm estate or factory. Next, biomass samples are prepared with the particle size less than 0.2mm by the means of crushing/milling and sieving process. The preparation of ionic liquids will be similar to the methods from the previous literature and the ionic liquids to be prepared are ethyl-methylimidazolium-acetate (EmimAc) and butyl-methylimidazolium-chloride (BmimCl). The biomass samples and ionic liquids will be dried using oven to make sure the effect of water on the subsequent dissolution process is minimized to as lowest as possible.

ii) Surface modification Treatment Of Biomass By Dissolution In Ionic Liquid

The biomass residue will mix with the ionic liquid and the mixture will be heated in the temperature range of 80-110^{o}C with constant stirring for 12-24hr in order to produce a homogeneous solution. The initial solid loading of biomass, heating temperature, time and the particles geometry greatly affect the dissolution process.

iii) Synthesis Of Biocomposite Gel

In order to prepare the biocomposite gel-type film, the homogeneous biomass-ionic liquid solution will be poured into a petri dish. Then, allow the solution to dry for several hours to synthesize a gel-type biocomposite polymer material.

iv) Fabrication of Hardboard

The dried polymeric material is put into specially-designed moulds and the desired shapes of biocomposites can be obtained by using the thermal/compression molding technique in the temperature range of 90-120^{o}C and the pressure range of 5-15MPa. The temperature and pressure are to be optimized so that the intrinsic lignin can be melted and acts as an internal binder by cross-linking mechanism. Then the dried material will be coagulated with water or ethanol which act as anti-solvent to remove the ionic liquid from the biocomposite and the final product is obtained.

3.1.2 Methodology (Changing of Final Year Project Title)

The biocomposite boards produced from OPF by using previous method is possess very weak structure. When it is mixed with high proportion of PKS, the biocomposite board structure is very strong. Yet, the production source of PKS from is not as much as OPF. Besides, the grinding process of PKS requires much more energy compared to OPF because it is much more harder compared to OPF. Thus, producing biocomposite boards using the mixture of OPF and PKS is the best combination rather than purely PKS. External binder (thermoplastic starch) is added to increase the adhesiveness between the OPF and PKS in the boards. The new methodology of biocomposite boards preparation is as below:

i) Preparation of Thermoplastic Starch (TPS)

Starch powder will first dissolve in ionic liquid before small amount of water is added to the mixture. The ratio of ionic liquid to starch to water is 0.3g:1g:0.2g. The mixture is then stirred and heated at 60^{o}C for 20 min to ensure thorough dissolve of starch in ionic liquid. The resultant mixture will then be left for one night to stabilize the mixture. 5 mixtures will be made which consist of starch mixing with 1-ethyl-3-methylimidazolium diethyl phosphate (EmimDEP), 1-butyl-3-methylimidazolium chloride (BmimCl), 1-ethyl-3-methylimidazolium acetate (EmimAc) and 1-butyl-3-

methylimidazolium trifluoromethane sulfonate (BmimTFMS) and one without ionic liquid as a reference experiment.

ii) Dissolution of TPS With Water

TPS is added with water until all the TPS is dissolved in the water. (Ratio of TPS to water is 1:9). This is to ensure a better mixing between the TPS and the biomass powder in the subsequent step. After the biomass powder (Ratio of TPS to OPF to PKS is 1:7:2) is added into the TPS, the mixture will be stirred until thorough mixing is achieved. This step is repeated for all of the TPS prepared before this.

iii) Thermal Compression Molding

The mold will be filled with the mixture of biomass and TPS and compressed by using spatula to ensure the space is enough to be filled with all the mixture produced. The inner and outer surface of the mold spacing is required to be rubbed with paraffin in order to achieve easier removal of the compressed samples and also the mold cap after the thermal compression process. The thermal compression molding machine will be adjusted so that the heating temperature is 200^{o}C and pressure of 26MPa. The thermal compression will carry out for 10 min and the mold is allowed to be cooled until the temperature achieves 50^{o}C under compression. The biocomposite boards produced will be removed from the mold spacing and keep in plastic containers.

iv) Properties Testing

The biocomposite boards will be tested using FTIR, DSC and TGA. The results obtained will be analyzed.

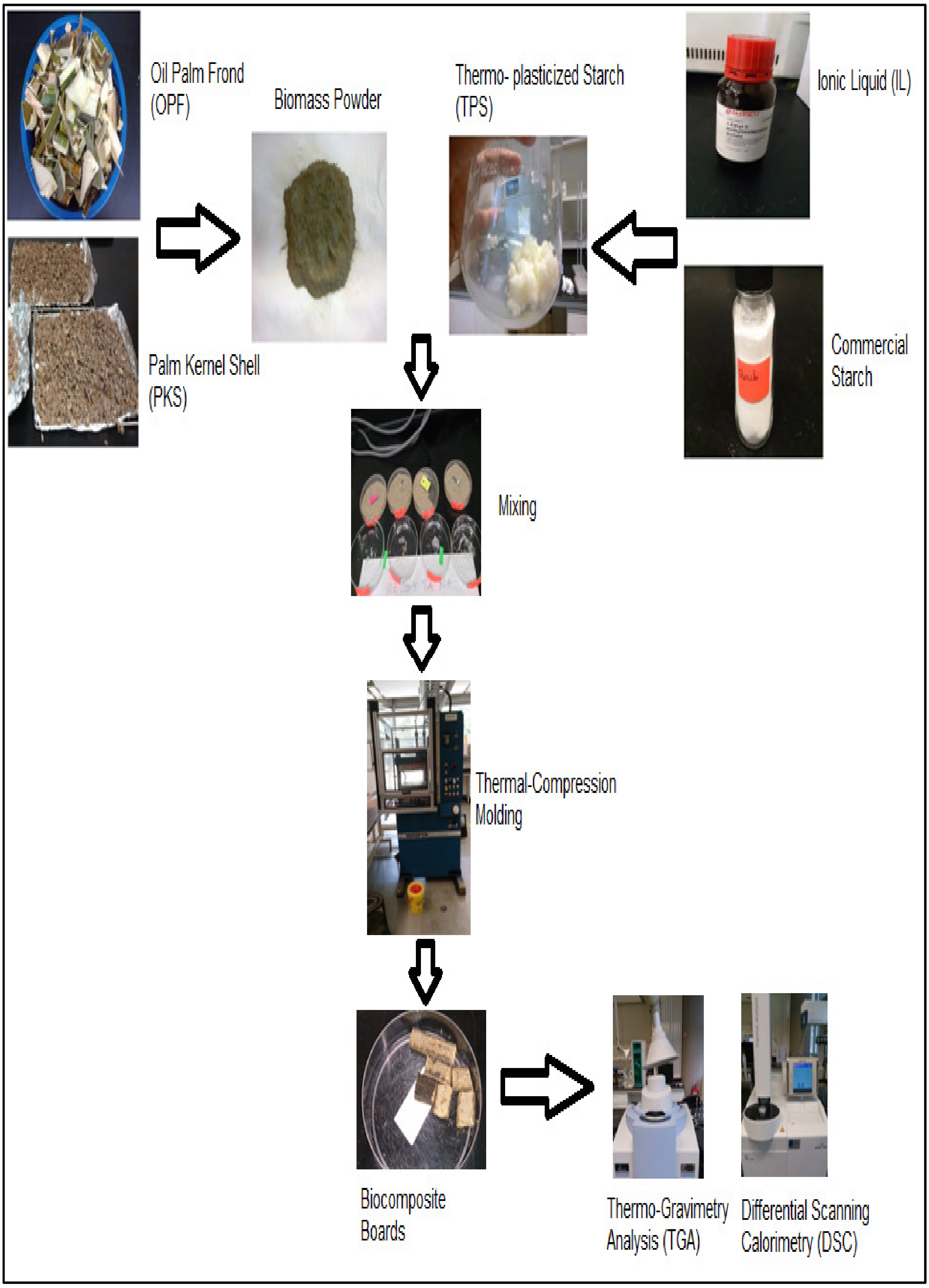


FIGURE 1: Process flow of the methodology.

3.2 KEY PROJECT MILESTONE

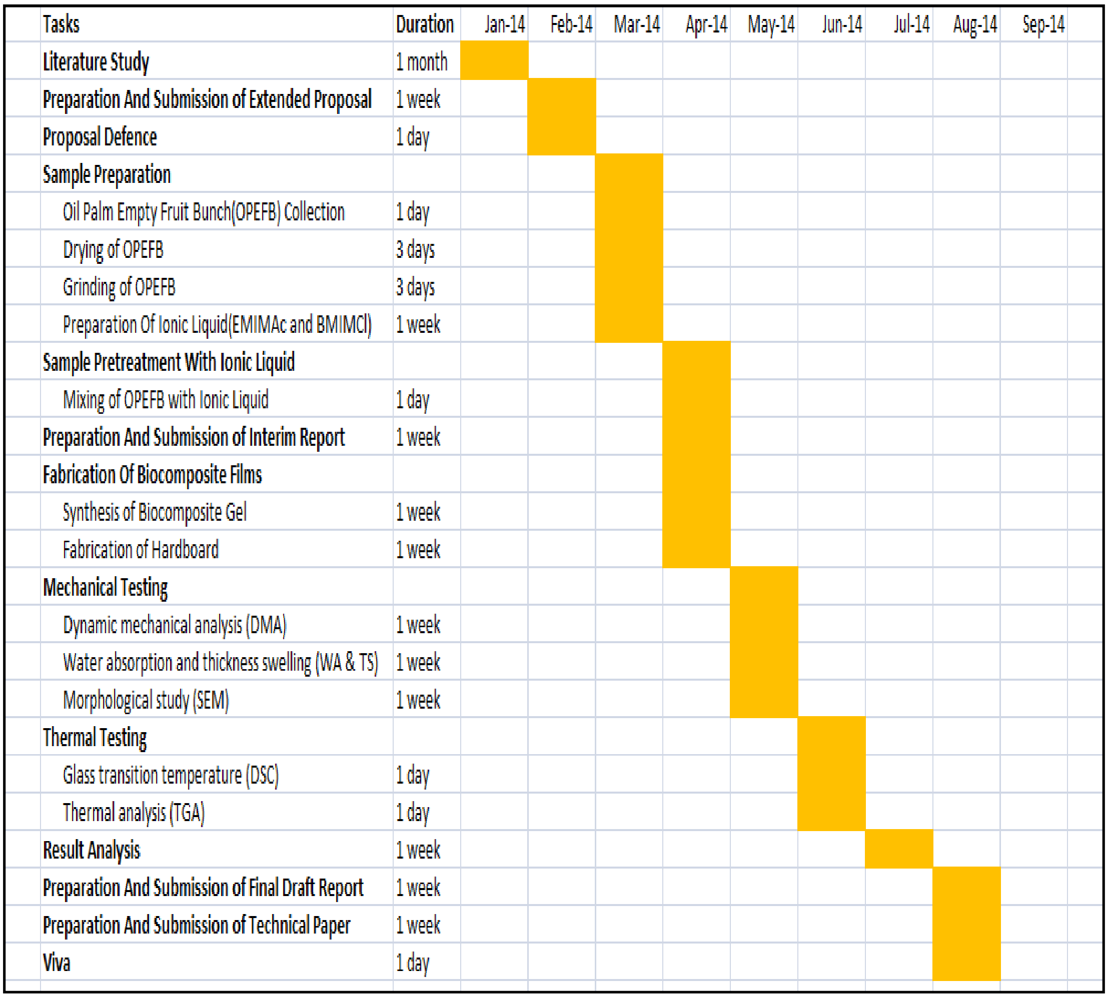
Key project milestone is a list of the main stages of progress throughout the final year project. It includes the targeted time to be accomplished together with the actual progress status of the respective stages. Each stage is to be accomplished according to the sequence which is listed in the following Table 2.

TABLE 2: Key project milestones.

Milestone	**Targeted Time**	**Status**
Sample Preparation (Grinding of Biomass)	March 2014	Completed
Sample Pretreatment With Ionic Liquid	March 2014	Completed
Fabrication of Biocomposite Boards	April 2014	Completed
Fabrication of Biocomposite Boards	May 2014	Completed
Properties Testing (FTIR, TGA, DSC)	June 2014	Completed
Result Analysis	July 2014	Completed

3.3 GANTT CHART

A Gantt chart had been created to make sure the project progresses as what had been planned and to check for the tasks to be accomplished. It is a more detailed version of the key project milestone as it includes the activities in each key milestone. With a Gantt chart, all the tasks can be performed in a more systematic way. The Gantt chart is as shown in the Figure 2.



Tasks	Duration	Jan-14	Feb-14	Mar-14	Apr-14	May-14	Jun-14	Jul-14	Aug-14	Sep-14
Literature Study	1 month	🟨								
Preparation And Submission of Extended Proposal	1 week		🟨							
Proposal Defence	1 day		🟨							
Sample Preparation				🟨						
Oil Palm Empty Fruit Bunch(OPEFB) Collection	1 day			🟨						
Drying of OPEFB	3 days			🟨						
Grinding of OPEFB	3 days			🟨						
Preparation Of Ionic Liquid(EMIMAc and BMIMCl)	1 week			🟨						
Sample Pretreatment With Ionic Liquid					🟨					
Mixing of OPEFB with Ionic Liquid	1 day				🟨					
Preparation And Submission of Interim Report	1 week				🟨					
Fabrication Of Biocomposite Films					🟨					
Synthesis of Biocomposite Gel	1 week				🟨					
Fabrication of Hardboard	1 week				🟨					
Mechanical Testing						🟨				
Dynamic mechanical analysis (DMA)	1 week					🟨				
Water absorption and thickness swelling (WA & TS)	1 week					🟨				
Morphological study (SEM)	1 week					🟨				
Thermal Testing							🟨			
Glass transition temperature (DSC)	1 day						🟨			
Thermal analysis (TGA)	1 day						🟨			
Result Analysis	1 week							🟨		
Preparation And Submission of Final Draft Report	1 week								🟨	
Preparation And Submission of Technical Paper	1 week								🟨	
Viva	1 day								🟨	

FIGURE 2: Gantt Chart of Final Year Project.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 PRETREATMENT OF OIL PALM BIOMASS WITH IL

The experimental part of the final year project has been started in the earlier March of FYP 1 because of the few reasons. Firstly, the feedstock of the experiment (OPEFB, OPF and PKS) which were collected from the nearby factory were available in the lab. Besides, in case the experiment does not yield good result, more time is available to analyze and study the results obtained. Possible reasons for the errors in the result is found out before revising and conducting new the experiment by changing different parameters until the good result is obtained. Finally the sample preparation, ionic liquid pretreatment, biocomposite boards preparations and properties testing consume a lot of time and thus all of the stages can be carried out more systematically when the experiment is started earlier.

The experiment was conducted using OPF initially because the OPEFB collected from the industry were attacked by the fungus. Thus, OPF was dried under the sun and oven for few days which were then grinded into powder form with size less than 0.25mm. Next, the OPF powder was mixed with ionic liquid at the weight ratio of 1:10 (OPF:IL). The initial IL used was 1-ethyl-3-methylimidazolium acetate. The mixture was stirred and heated for one hour. The obtained mixture was too liquid to be proceed for thermal compression. Thus, another sample was prepared again with the new weight ratio of 6:4 (OPF:IL). The mixture obtained was still in the powder form but more sticky than the raw OPF powder. Thus, the sample proceed with the thermal compression. The first trial operating temperature and pressure was 200^{o}C and 20MPa with the compression time of 20min. The result was failed because the whole sample was totally burned out into black charcoal. Thus, the experiment was repeated with different process conditions of temperature, pressure and thermal compression time in order to get the optimum conditions for the best board properties.

Generally, the biomass pretreatment with IL are similar with the pretreatment time of 1 hr±10min, heating temperature of 100°C-140°C and the stirring rate of 700rpm. The thermal compression involve the use of the mold of small rectangular shape instead of other bigger shape like dog-bone shape (for ASTM standard) because other shapes required more amount of biomass to be filled and it would be a wastage when the experimental work is still in the process of determining the optimum thermal compression conditions. The summary of the result of thermal compression molding was as shown in Table 3. Meanwhile, the IL used was changed to other IL because the available amount of IL mentioned previously was not sufficient to carry out new experiment. Besides, it is a wastage to carry out trials with highly specialized IL. The second batch of OPEFB was also available in the lab and thus sample preparation was done to produce OPEFB powder. Pretreatment of IL and thermal compression molding were also done with the OPEFB powder but the main focus is put on OPF since the experiment was started with OPF. It was found out that both OPF and OPEFB will be darker in color and swell after pretreated with IL. But there are not much difference in color between the raw biomass and regenerated biomass. Apart from that, experiment was also conducted with raw palm kernel shell (PKS) powder which has the same particle size as OPF and OPEFB grinded before this. Biocomposite boards with greatly improved mechanical strength were obtained using PKS especially when raw OPF was mixed with PKS.

TABLE 3: Summary of the results obtained after thermal compression molding.

Experiment	Process Conditions	Result	Discussions
1	Feedstock: OPF+IL Temperature:200°C Pressure: 20MPa Time: 20min	The whole sample burned out. A black charcoal-like substance obtained.	Possible reason due to the cooling of thermal compressed sample with pressure removed. This causes the decomposition of sample to occur. Other reason may be the presence of IL causes the lignin and cellulose to decompose at much lower temperature.

2	Feedstock: Raw OPF Temperature:140°C Pressure: 20MPa Time: 20min	The whole sample burned out. A black charcoal-like substance obtained.	Possible reason due to the cooling of thermal compressed sample with pressure removed. This causes the decomposition of sample to occur. Other reason may be the presence of IL causes the lignin and cellulose to decompose at much lower temperature.
3	Feedstock: OPF+IL Temperature:200°C Pressure: 20MPa Time: 10min	The whole sample burned out but still had a structure. A black charcoal-like substance obtained.	Possible reason due to the cooling of thermal compressed sample with pressure removed. This causes the decomposition of sample to occur. Other reason may be the presence of IL causes the lignin and cellulose to decompose at much lower temperature.
4	Feedstock: Raw OPF Temperature:200°C Pressure: 20MPa Time: 10min	The color of thermal compressed sample similar to raw OPF and easily revert into powder form.	No or limited amount of cross-linking between lignin and cellulose at this process condition.
5	Feedstock: raw OPF Temperature:170°C Pressure: 20MPa Time: 9min	The color of thermal compressed sample similar to raw OPF and easily revert into	No or limited amount of cross-linking between lignin and cellulose at this process condition.

		powder form.	
6	Feedstock: Regenerated OPF Temperature: 180°C Pressure: 20MPa Time: 20min	The color of thermal compressed sample similar to raw OPF and easily revert into powder form.	No or limited amount of cross-linking between lignin and cellulose at this process condition. Other possible reason is due to some of the lignin and cellulose have been washed out from the sample during regeneration of IL.
7	Feedstock: regenerated OPF Temperature:195°C Pressure: 20MPa Time: 10+10min	The color of thermal compressed sample similar to raw OPF and easily revert into powder form.	No or limited amount of cross-linking between lignin and cellulose at this process condition. Other possible reason is due to some of the lignin and cellulose have been washed out from the sample during regeneration of IL.
8	Feedstock: raw OPF Temperature: 180°C Pressure: 20MPa Time: 20min	The color of thermal compressed sample similar to raw OPF and easily revert into powder form.	No or limited amount of cross-linking between lignin and cellulose at this process condition.
9	Feedstock: raw OPF Temperature:195°C Pressure: 20MPa Time: 10min	Structure of biocomposite board is obtained.	Partial cross-linking between lignin and cellulose start to occur at this temperature.
10	Feedstock: raw OPF	The thermal compressed	Partial cross-linking between lignin and cellulose start to

	Temperature:200°C Pressure: 20MPa Time: 20min	sample has darker brown color compared to raw OPF. Structure of biocomposite board is obtained.	occur at this temperature. Small amount of lignin or cellulose started to decompose which account for the darker color of thermal compressed sample.
11	Feedstock: raw OPF Temperature:210°C Pressure: 20MPa Time: 20min	The thermal compressed sample has darker brown color compared to raw OPF. Structure of biocomposite board is obtained.	Partial cross-linking between lignin and cellulose start to occur at this temperature. Small amount of lignin or cellulose started to decompose which account for the darker color of thermal compressed sample.
12	Feedstock: raw OPF Temperature: 240°C Pressure: 20MPa Time: 10min	The thermal compressed sample has darker brown color compared to raw OPF. Structure of biocomposite board is obtained but easy revert to powder form.	Possibility that lignin and cellulose start to decompose at this temperature which affect the cross-linking process.
13	Feedstock: Regenerated OPF Temperature:210°C	The thermal compressed sample has	Possible reason is due to some of the lignin and cellulose have been washed

	Pressure: 20MPa Time: 20min	darker brown color compared to raw OPF. Structure of biocomposite board is obtained. Easier to revert to powder form compared to trial with raw OPF.	out from the sample during regeneration of IL which yield a weaker structure compared to raw OPF.
14	Feedstock: OPF+IL Temperature:195°C Pressure: 20MPa Time: 10min	A sticky structure is obtained with the surfaces burned out. The structure easily reverts to powder.	Possible reason may be the presence of IL causes the lignin and cellulose to decompose at much lower temperature.
15	Feedstock: Raw PKS Temperature:195°C Pressure:26.67MPa Time: 20min	A structure with hardness greatly improved compared with raw OPF but brittle was obtained. The color remained the same as grey color.	The color remained as raw PKS means no burn(decomposition) of the sample. PKS has great mechanical strength but brittle. Possible reason that PKS contains higher cellulose composition compared to OPF since cellulose provides mechanical strength.
16	Feedstock: Raw OPF+ Raw PKS(Ratio:1:1)	A black color-structure with excellent	The black color is mainly due to the burned OPF. The mixture of raw OPF and raw

	Temperature:210^{o}C Pressure:26.67MPa Time: 10min	strength and less brittle was obtained.	PKS provides much better mechanical strength. Possible reason that OPF contains higher lignin composition which binds the cellulose and makes the structure to be less brittle.

From the results obtained, it can be observed that the raw OPF especially in the presence of PKS will yield better biocomposite boards compared to both the pretreated OPF and regenerated OPF. This result deviates from the theoretical discussion whereby the pretreatment with IL will breaks the internal binder lignin to ease the cross-link formation to bind the cellulose during the thermal compression molding process which means the pretreated OPF should yield a better biocomposite structure compared to the raw OPF. The possible reason for this is that the cooling process of the pretreated OPF was being done with the pressure relieved which may cause decomposition of the sample. On the other hand, the deviation of result obtained from regenerated OPF might be due to that most of the lignin had been washed out by the water from the OPF during regeneration process. Thus, by means of visual inspection, raw OPF and PKS mixture will give the best biocomposite board structure. In terms of processing parameters, the optimum temperature is found to be 200^{o}C, pressure of 26Mpa, and thermal compression time of 10min.

4.2 PRETREATMENT OF BIODEGRADABLE BINDER WITH IL

Due to the time limitation, the FYP will only focus on OPF and PKS only. In order to enhance the binding strength between OPF and PKS, external binder is used. Starch is a good binder but transforming it into thermoplastic starch (TPS) will improve its binding strength. Producing TPS by using water alone as the plasticizer of starch will yield hard particle of TPS. This is because crystallization occur upon water addition to the starch. From the previous researches, addition of IL can yield good TPS. TPS were prepared by mixing starch powder with IL and water. The mixture is prepared with 0.3g of IL, 1g of starch and 0.2g of water. The mixture is stirred with heating at 70°C until a thorough mixture is obtained before leaving the mixture for overnight. The observations of TPS obtained is as the table below and the photos of the TPS prepared is included in the appendices.

TABLE 4: Summary of the results obtained by visual inspection for TPS preparation.

IL	Observations
EmimDEP	Gel-like gummy substance is obtained upon mixing. Less gel-like upon left overnight.
BmimCl	Gel-like gummy substance is obtained upon mixing. Less gel-like upon left overnight.
EmimAc	Gel-like gummy substance is obtained upon mixing. Less gel-like upon left overnight.
Water (reference)	Gel-like gummy substance is obtained upon mixing. Hard crystal is obtained upon left overnight.
BmimTFMS	Worst dissolution power of the IL. Disperse particles was obtained. Hard particle is obtained upon left overnight.

The TPS obtained were dissolved in water under heating at 60°C before mixing with the biomass. This is to ease the mixing of TPS with biomass. The ratio of mixing for TPS: water is 1: 9. The ratio of TPS to biomass(OPF:PKS) is 10:70:20. This is followed by thermal compression molding. The mold filled with the resultant biomass is first heated at 140°C for 40 min before thermal compression (200°C, 26Mpa) for 10 min in order to remove the water content from the biomass. The results of the biocomposite boards obtained is as below and the photos of the biocomposite boards produced is included in the appendices.

TABLE 5: Summary of the results by visual inspection after thermal compression molding.

Types of IL used in TPS preparation	Observation on the biocomposite board
EmimDEP (1)	Board with very hard structure and very difficult to break apart.
BmimCl (2)	Board with very hard structure but not as compact as the board produced using EmimDEP.
EmimAc (3)	Board with very hard structure. The structure is better than that of using BmimCl but not as good as that of using DEP.
Water (4)	Board with very hard structure comparable with the one produced with EmimDEP. The structure slightly get burned.
BmimTFMS (5)	Board with weakest structure, easiest to break into fragments.

* The each IL is assigned with a number at the side which will be used to label the spectra in Figure .

Generally, all of the biocomposite boards produced with TPS added are more compact, strong and not easily revert into powder. This shows that TPS binds the OPF with the PKS in the biocomposite strongly. From the observations above, TPS made with EmimDEP gives the best binding forces for the biomass components.

4.3 TESTING WITH FTIR

Fragments where obtained from the biocomposite boards produced for FTIR testing to determine the hydrogen bonding within the biocomposite boards. The samples were put into glass canisters and sent to the Centralized Analytical Lab (CAL) of UTP for FTIR testing. In-situ Fourier Transform Infra Red Spectrometer with High Vacuum Purge System (In-situ FTIR-HVPS) machine is used for the testing. The spectra were recorded at the wavenumber range of 400-4000cm^{-1} and resolution of 4cm^{-1}. 5 spectra were generated for the 5 samples as shown below. Each peak represents the presence of particular functional group.

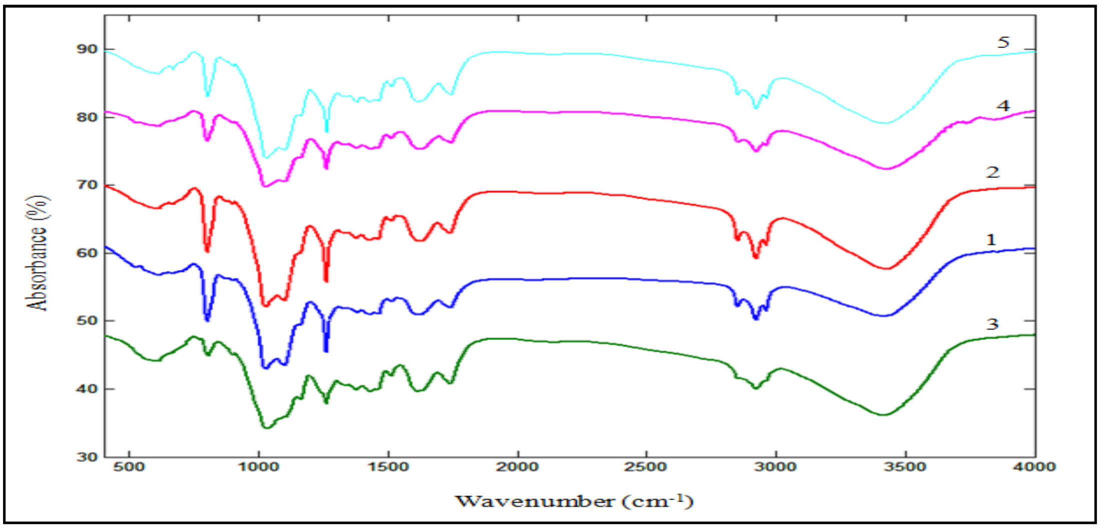


FIGURE 3: Absorption spectra of the samples.

In order to have a strong structure of biocomposite, the OPF and PKS must be able to form bonds. The main bond to be formed between the components is hydrogen bond because the main composition of the components consists of cellulose, starch and lignin which contain O-H group. The bond formation mechanism occurs between imidazolium salt anion and the proton in the hydroxyl group. The inter-hydrogen bonding formation may cause bond stretch to the O-H group. The broadening and shifting to lower wave number region of the region corresponding to O-H group means there are strong hydrogen bonding formation between the components. According to Abdulkani et al., inter-hydrogen bond formation can be detected by few characteristics in the FTIR spectra. Firstly, the shifting of band which corresponding to C-H bond stretching (wave number 2900 cm^{-1}) to higher wave number value and reduction in the peak intensity account for the increase in amorphous structure in the biocomposite. Inter-hydrogen bonding induce amorphous structure because the initial intra-hydrogen bonding within the individual component is disrupted and broken to form new bond with other component. Next, the intensity of the adsorption band (wave number 1500-899cm^{-1}) decreases. Thirdly, the decrease in the band (wave number 1430 cm^{-1}) intensity which accounts for the symmetric CH_2 vibration means more hydrogen bond formation.

From the spectra obtained above, with reference to the reference spectra of Figure 29 which is the biocomposite board produced with TPS without pretreatment with IL (%T for band around 2900cm^{-1} is 63%, %T for band 1500-899cm^{-1} is 57.5%-63.5%, %T for band 1430cm^{-1} is 63%), all other spectra possess the three phenomena discussed above with the exception of sample containing BmimTFMS. The summary of data comparison of the spectra is shown in the following table. The result shows that all samples except sample 5 (BmimTFMS), possess lower absorption intensity for the specific region discussed above. This means that biocomposite boards produced with TPS pretreated with IL (except BmimTFMS) will have more inter-hydrogen bond formation. This can be explained by the presence of IL which induce the formation of inter-hydrogen bonding between the OPF and PKS. On the other hand, the spectra shows that BmimTFMS will not induce inter-hydrogen bond formation which matches the observation made during TPS preparation whereby BmimTFMS has the lowest dissolution power of starch. With the intensity the lowest among others, the biocomposite board produced with TPS pretreated with EmimAc possess the most inter-hydrogen bonding which means the strongest biocomposite board. This is followed by EmimDEP and BmimCl.

TABLE 6: Comparison of the data of the spectra of FTIR testing.

Sample	%A for band around 2900cm^{-1}	%A for band 1500-899cm^{-1}	%A for band 1430cm^{-1}
1	50	42-55	51
2	62	52-68	62
3	40	34-45	40
4	75	70-79	75
5	82	74-87	82

4.4 TESTING WITH TGA

The biocomposites produced were tested using TGA in order to analyze the components contained in the biocomposites as well as the thermal stability of the biocomposite boards. The testing was conducted using Thermogravimetry-mass spectrometer (TG-MS) model STA 6000 Perkin Elmer. The samples were pressed into powder form before putting into the TGA testing containers. The amount put should be within the range of 2g to 5g. The samples were heated from 50°C to 800°C with the heating rate of 30°C per minute. The results were recorded in percent weight lost as a function of temperature. A sudden large decrease in weight signify that there is a component undergoes decomposition. It is also possible that there are more than one component decomposes at the same time whereby in this case, the decomposition temperature of the components are very similar. Figure 4 shows the TGA curve of the 5 samples.

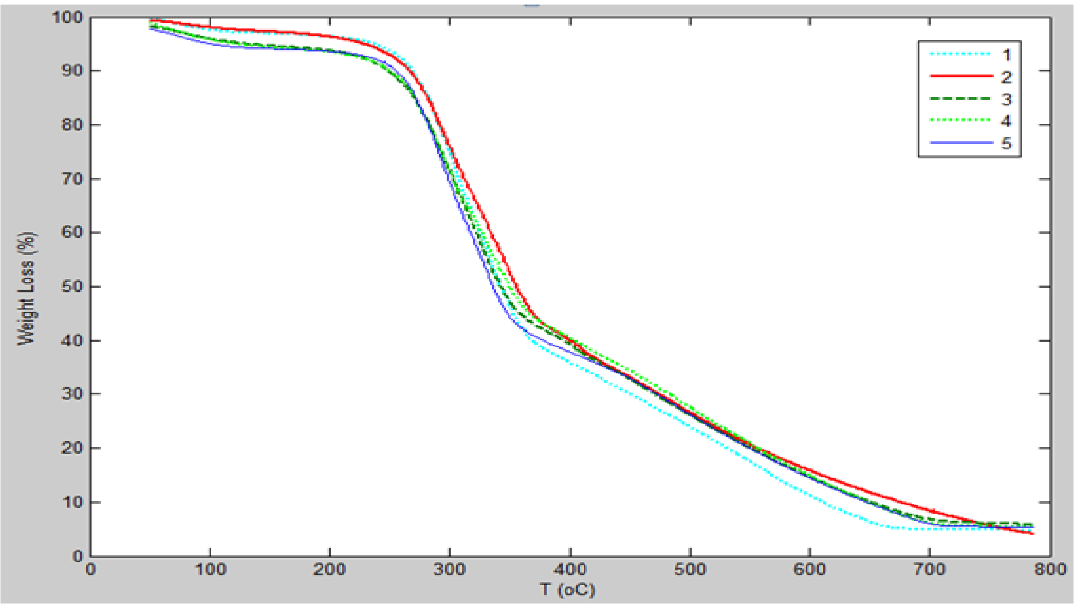


FIGURE 4: The TGA curve of the 5 samples.

From Figure 4, it can be seen that the TGA curves are very similar for all 5 samples, that is, all of the curves contain one less steep slope and two steep slopes. This means that there are two main decompositions in the samples during the heating which means there are two decomposition temperatures. The first slope starts before 100°C and this is attributed to the water removal from the samples. The first steep slope is steeper than the second steep slope. This means that the decomposition of the first decomposed component occur at a faster rate. The first decomposition temperature is between 260°C to 280°C whereas the second decomposition temperature is between 360°C to 390°C.

From the literature survey, the decomposition of cellulose will begin around 250°C (Randriamanantena, T. et al., n. d.), starch will range from 280°C to 350°C with most probably 300°C (Mano, J. F., Koniarova, D., & Reis, R. L., 2003) and lignin will have slower and broader decomposition temperature which range from 200°C to 500°C with the main decomposition starts at 400°C (Brebu, M., & Vasile, C., 2009). Thus, it is proposed that the first steep slope represent the decomposition of starch and cellulose and the second steep slope accounts for the lignin decomposition.

The significance of TGA test is to show the thermal stability and the components contained in the biocomposite boards. Thus, it can be concluded that the biocomposite boards are thermally very stable until very high temperature (>250°C). This means that it can be safely used in high temperature applications. Besides, It can also be concluded that the biocomposite boards contain 3 main components: cellulose, starch and lignin.

4.5 TESTING WITH DSC

The biocomposite boards were tested with DSC in order to determine the thermal stability of the biocomposite boards. The DSC test was carried out using the Differential Scanning Calorimeter model DSC 1 Mettler Toledo. The tests were conducted under the nitrogen gas atmosphere using the sample weight of range 5-10mg. Each sample was first heated isothermally at 130°C for 30min to remove the moisture contained in the sample. This is then followed by the cooling to -80°C because the glass transition temperature of ionic liquid used in the project (EmimDEP, BmimCl, EmimAc, BmimTFMS) range below -50°C. The sample was hold at this temperature for 10 min. After that, it was being heated to 200°C followed by cooling to -80°C again. Both the cooling rate and heating rate are 10°C per min. In short, the whole process consists of 5 stages and each stage is labeled from 1 to 5 accordingly as shown in the following diagram (Figure 5). In this discussion, only stage 2, 4 and 5 will be included because the isothermal section (stage 1 and stage 3) do not give significant meaning to the testing. Throughout the whole process, the sample pan is required to maintain the same temperature as the reference pan. The extra heat released or absorbed by the sample pan in the calorimeter in order to maintain the same temperature as the reference pan is recorded. The DSC curve is a graph of heat absorbed or released against the temperature.

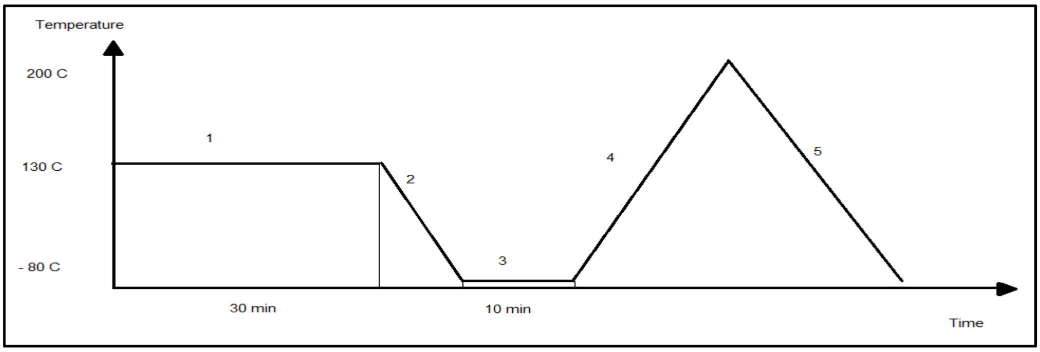


FIGURE 5: Temperature profile of DSC test.

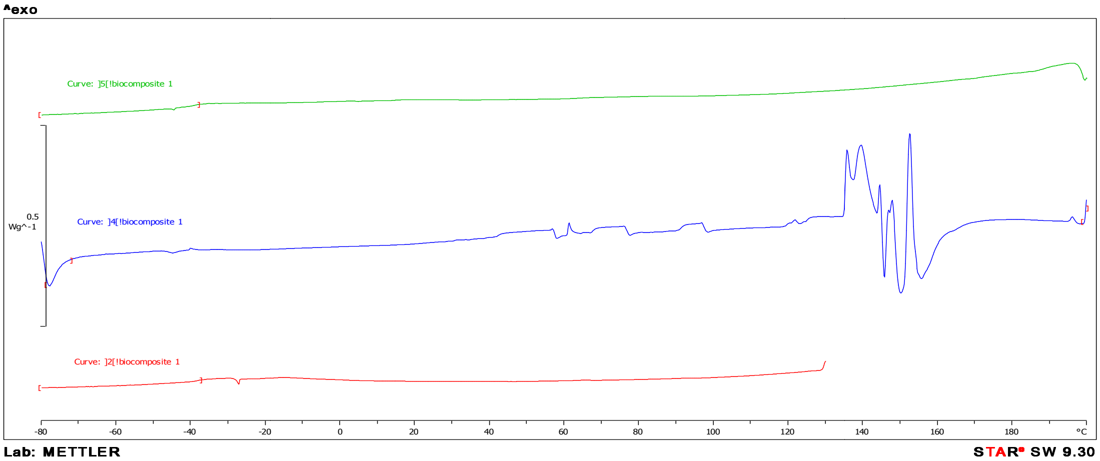


FIGURE 6: DSC curve of biocomposite board 1. (with IL EmimDEP)

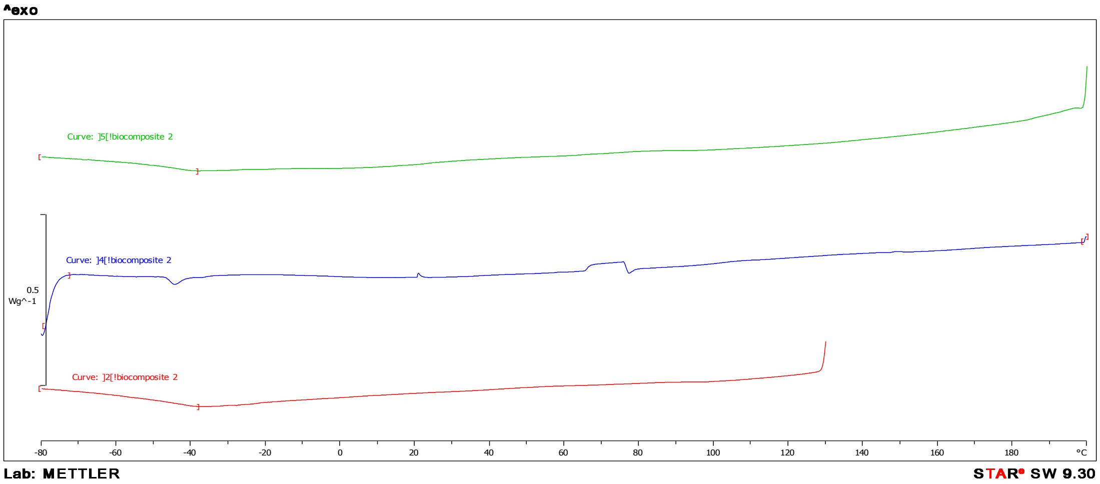


FIGURE 7: DSC curve of biocomposite board 2. (with IL BmimCl)

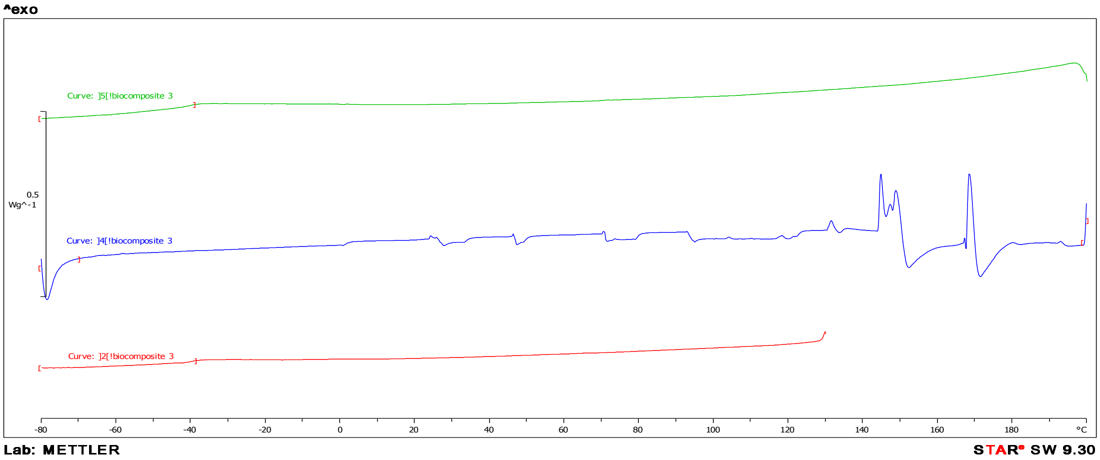


FIGURE 8: DSC curve of biocomposite board 3. (with IL EmimAc).

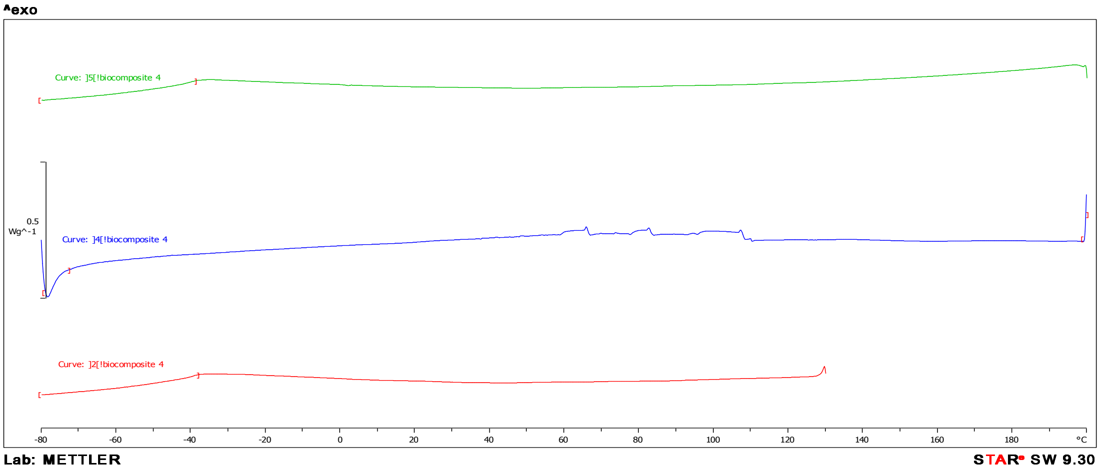


FIGURE 9: DSC curve of biocomposite board 4. (reference sample with no IL).

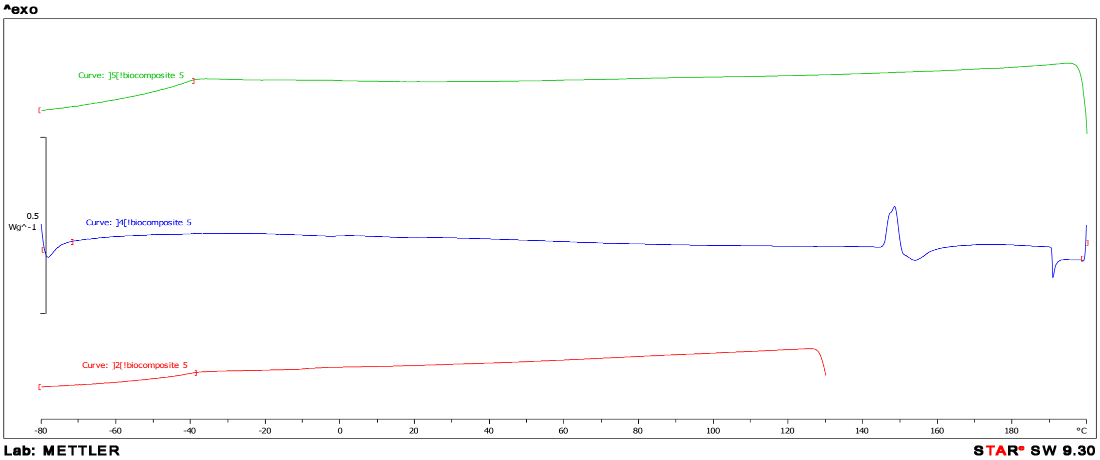


FIGURE 10: DSC curve of biocomposite board 5. (with IL BmimTFMS).

The upmost curve (green color line) represents the DSC curve of the cooling process from 130^{o}C to -80^{o}C; the middle curve (blue color line) represents the DSC curve of the heating process from -80^{o}C to 200^{o}C; the most bottom curve (red color line) represents the cooling process from 200^{o}C to -80^{o}C. Generally, both cooling curves (curve 2 and 5) do not contain obvious peak. Basically, the DSC test is used to determine the thermal stability of the biocomposite boards. Upward peak signifies crystallization whereas downward peak signifies glass transition temperature or melting point. Since the biocomposite boards consist of naturally occurring polymers (cellulose, starch, lignin) whereby the melting point is very high (>200^{o}C), the DSC curve 4 should yield a relatively straight line. Yet, the results obtained contain random peaks. The possible reason for this phenomena is that there are background interference which was not being cleared before running the test on the samples. Another possible reason is that the biocomposite boards might be polluted by the impurities left in the mold from the previous compression molding.

Thus, it is recommended that the DSC test should be repeated by first clearing the background interference before heating the samples. If the random peaks still exists, new samples should be prepared again with ensuring the thermal compression mold is clean before putting the biomass powder into the mold.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

Synthetic polymer products have many disadvantages which are the consumption of non-renewable resources, disposal problem and non-environmental friendly production process. On the other hand, biopolymers are renewable, bio-degradable and produced by environmental friendly process. Literatures about the ionic liquid as a solvent for the binder (starch) to prepare biocomposites have been studied and reviewed. Among the parameters that affect the biocomposite properties includes the types of biopolymers used in the biocomposite blend, the composition of the biopolymers in the blend, the types of coagulating agents for solvent removal and so on.

From the experiment done, the pretreatment of IL on natural polymers seems not to improve the binding force between the natural polymers. The raw biomass generally can produce a better structure with better mechanical strength compared to the biomass pretreated with IL. Since the results obtained from the experiment do not match entirely with the theories obtained from the literatures and also the raw PKS and OPF mixture produces outstanding result on the structure and mechanical strength, the project focuses on the preparation and properties testing of biocomposite using OPF and PKS with TPS pretreated with IL. From the results obtained at the earlier stage of the project, it can be concluded that the best thermal compression temperature and pressure as well as the thermal compression time is 200^{o}C, 26Mpa and 10min respectively.

The result of the FTIR shows that the presence of IL will trigger the formation of inter-hydrogen bonding between the components contained in the biomasses with the exception of BmimTFMS. The result from TGA shows that the biocomposite boards are thermally stable until very high temperature (>250^{o}C). This means the boards are safe to use under high temperature applications. On the other hand, there is no obvious result from DSC to show the biocomposite boards are thermally stable.

It is recommended that more experiment and research should be done on the contradicting results obtained in the project (pretreatment of raw biomass powder on the improvement of the strength of the biocomposite boards), the effect of the types and amount of binders used and the effect of different combinations of biomass. In addition, the testing on mechanical strength of the biocomposite boards, water absorption and thickness swelling of the boards are required in order to comply with international standards like ASTM (American Society of Testing And Materials), JIS (Japanese Industrial Standards) and so on. This can improve the economic potential of the biocomposite boards.

In conclusion, more research are still need to be done in this field as the current data are still not matured enough to produce the biocomposites with good qualities in large scales. Thus, it is recommended that more effort to be put on the research of different ionic liquids and discover more biopolymers that can produce biocomposites with properties which are comparable with the synthetic polymers. Only that, biocomposites can substitute the synthetic polymer products and environmental pollution can be reduced.

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APPENDICES

FIGURE 11: IL:1-ethyl-3-methylimidazolium acetate.

FIGURE 12: Raw OPF powder.

FIGURE 13: Raw OPEFB powder.

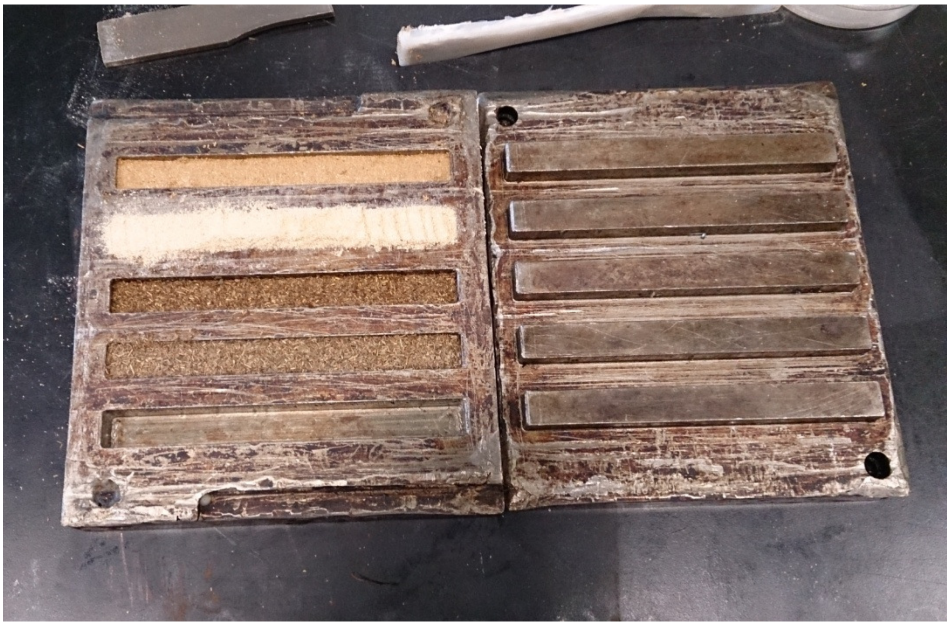

FIGURE 14: Thermal compression molding.

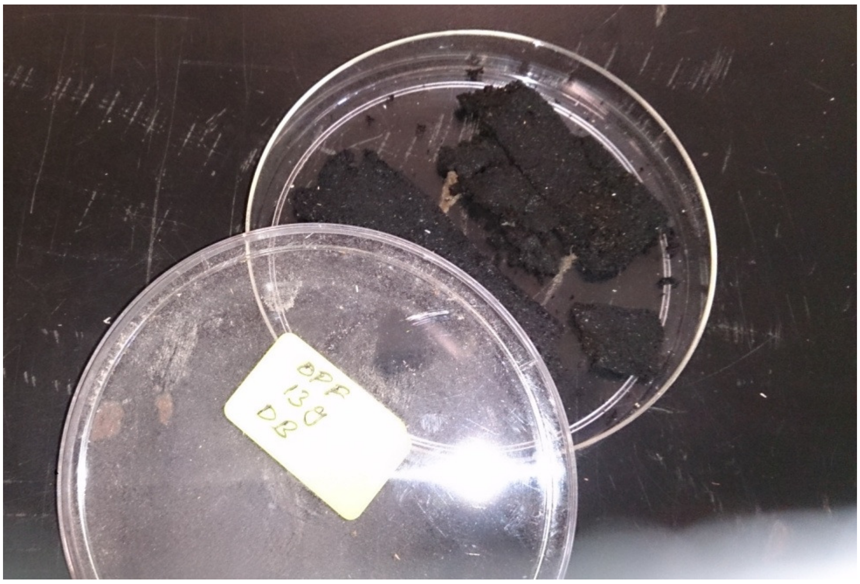

FIGURE 15: Result of first trial- OPF/IL turned into black charcoal at temperature 200°C, pressure 20MPa, time 20min.

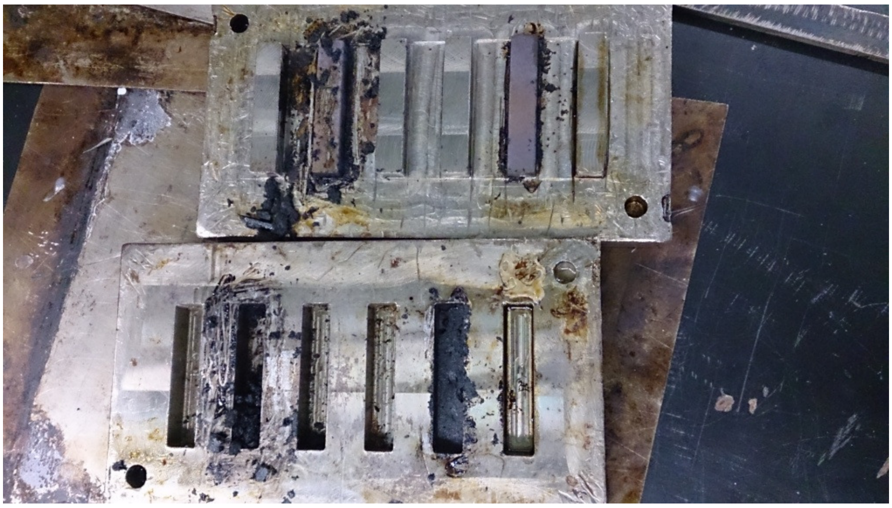

FIGURE 16: Result of first trial- OPEFB/IL turned into black charcoal at temperature 200°C, pressure 20MPa, time 20min.

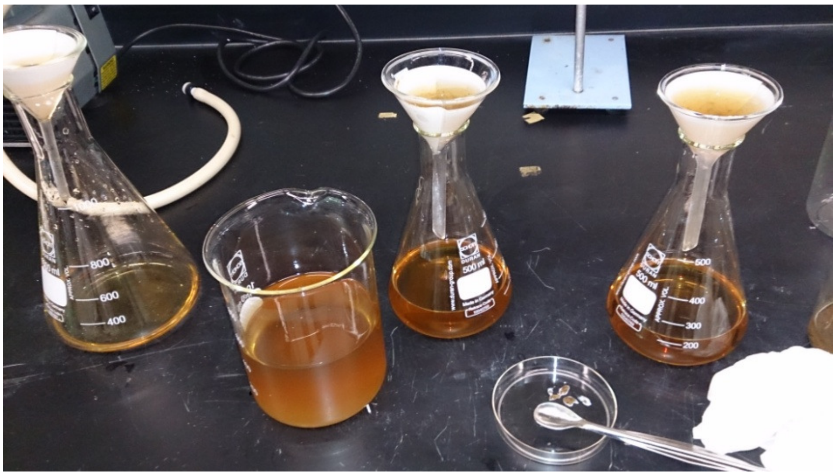

FIGURE 17: Regenerating OPEFB using manual filtration.

FIGURE 18: Regenerating OPF using vacuum filtration.

FIGURE 19: Regenerated IL solution.

FIGURE 20: Biocomposite films made from raw OPF under temperature 210^{o}C, pressure 20Mpa, compression time 10min.

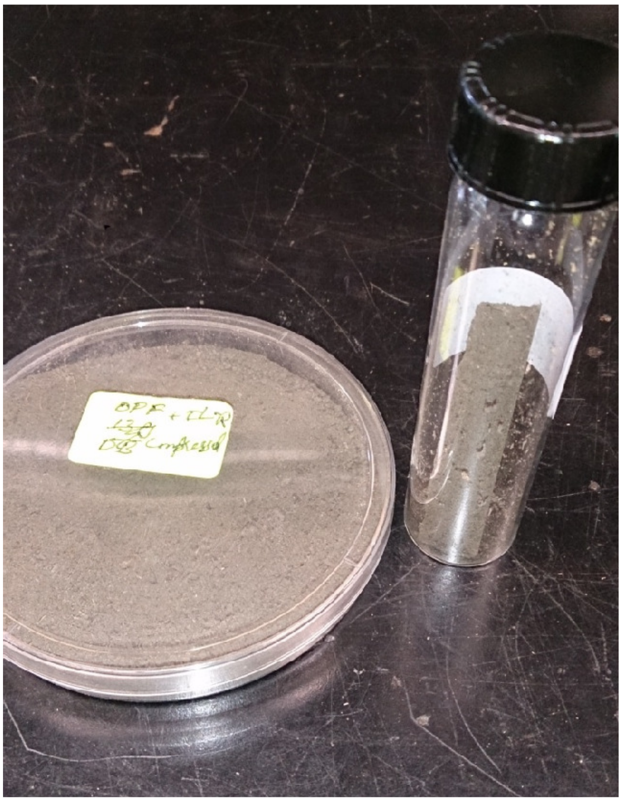

FIGURE 21: Raw PKS and thermal compressed PKS.

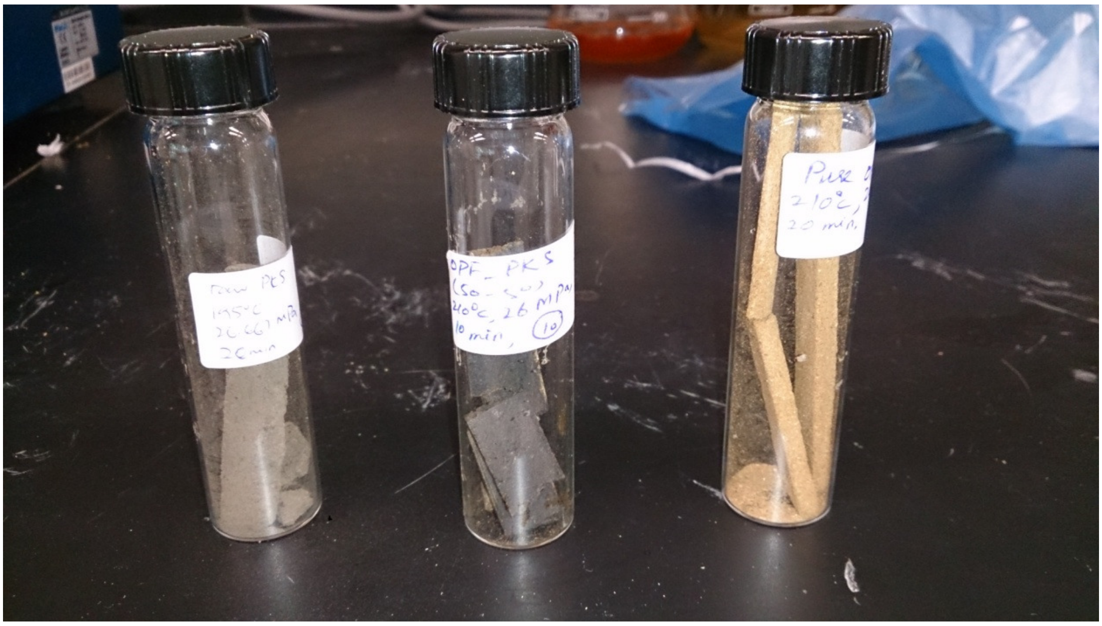

FIGURE 22: Starting from left: Biocomposite boards produced using raw PKS, raw OPF-raw PKS and raw OPF.

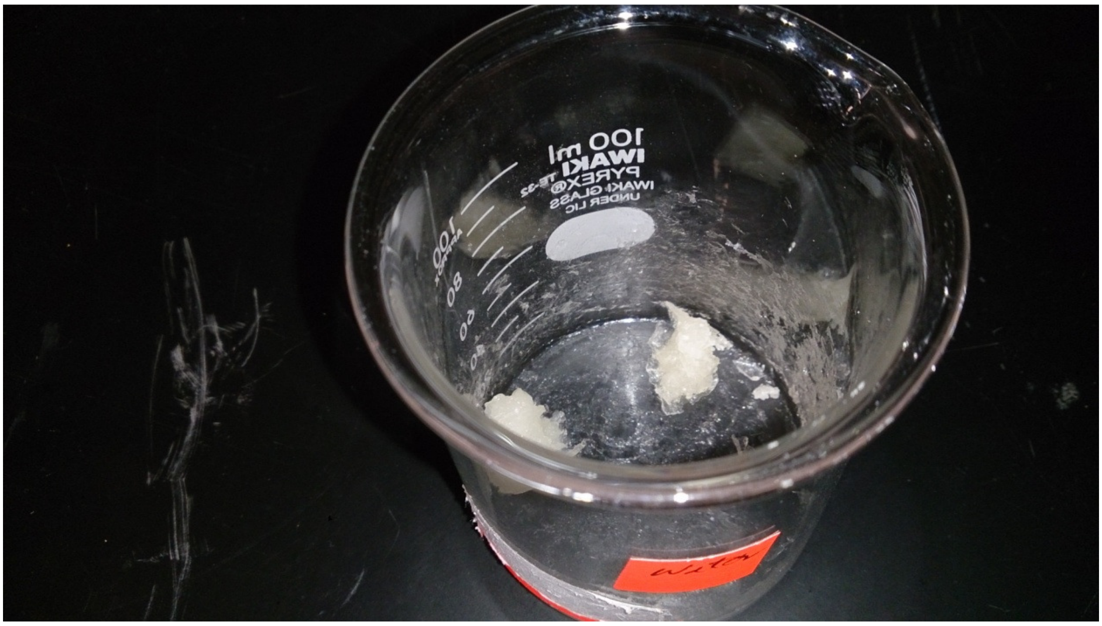

FIGURE 23: TPS prepared by dissolving starch in water.

FIGURE 24: TPS pretreated with IL after left overnight.

FIGURE 25: (Left) Biomass before thermal compression. (Right) Biocomposite boards produced after thermal compression. (Mold column from left to right: EmimDEP, BmimCl, EmimAc, Water, BmimTFMS).

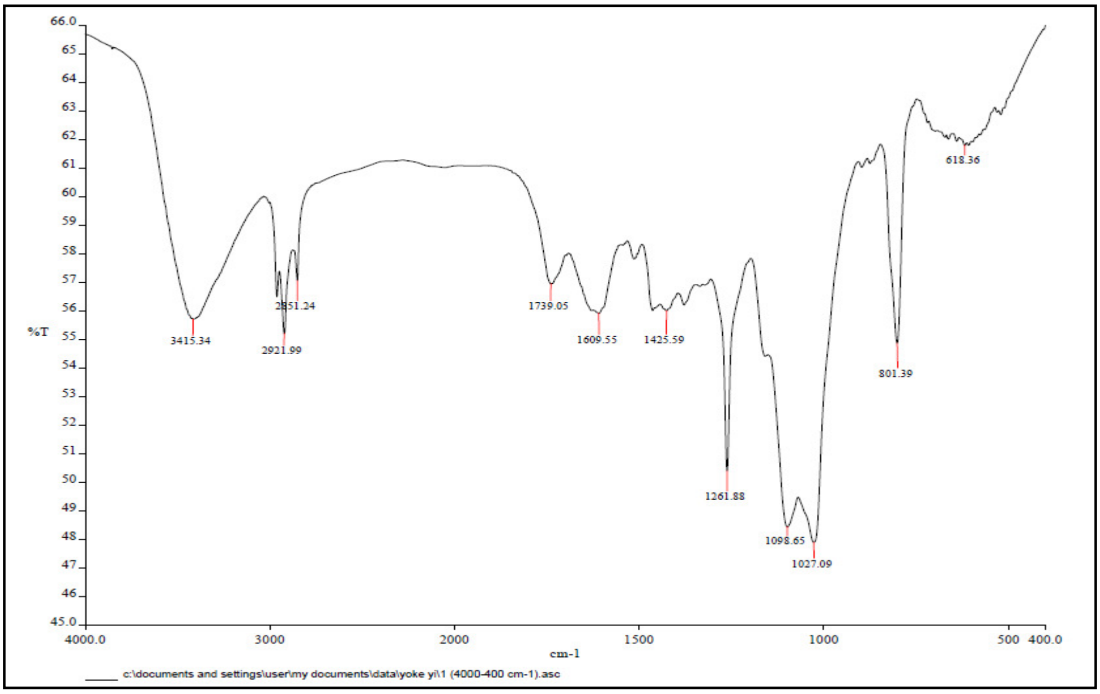

FIGURE 26: FTIR spectra of biocomposite produced with TPS pretreated with EmimDEP.

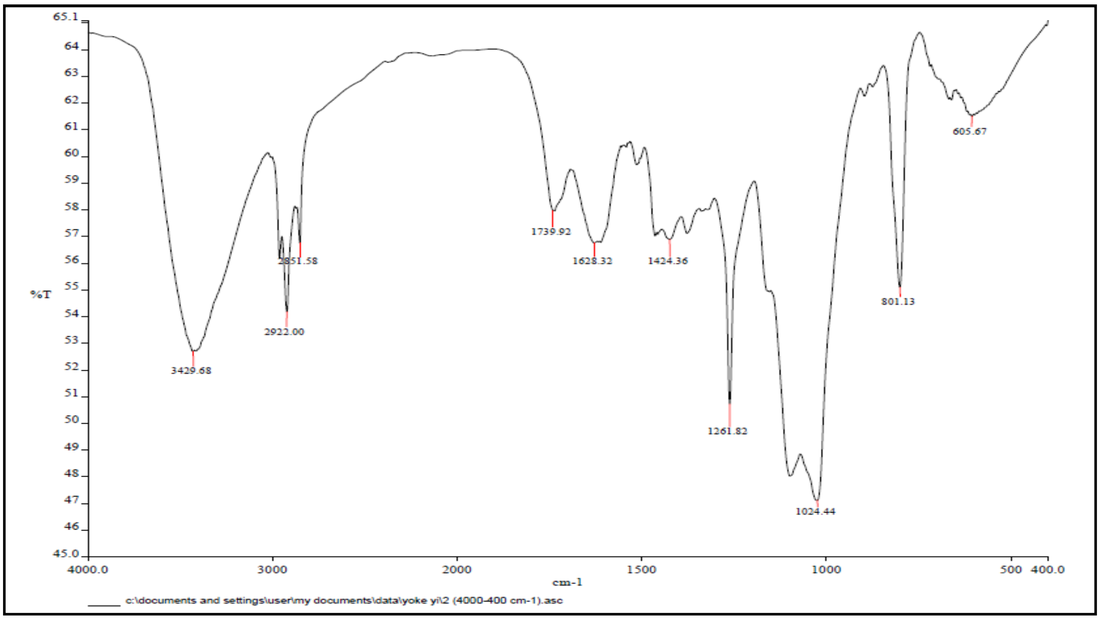

FIGURE 27: FTIR spectra of biocomposite produced with TPS pretreated with BmimCl.

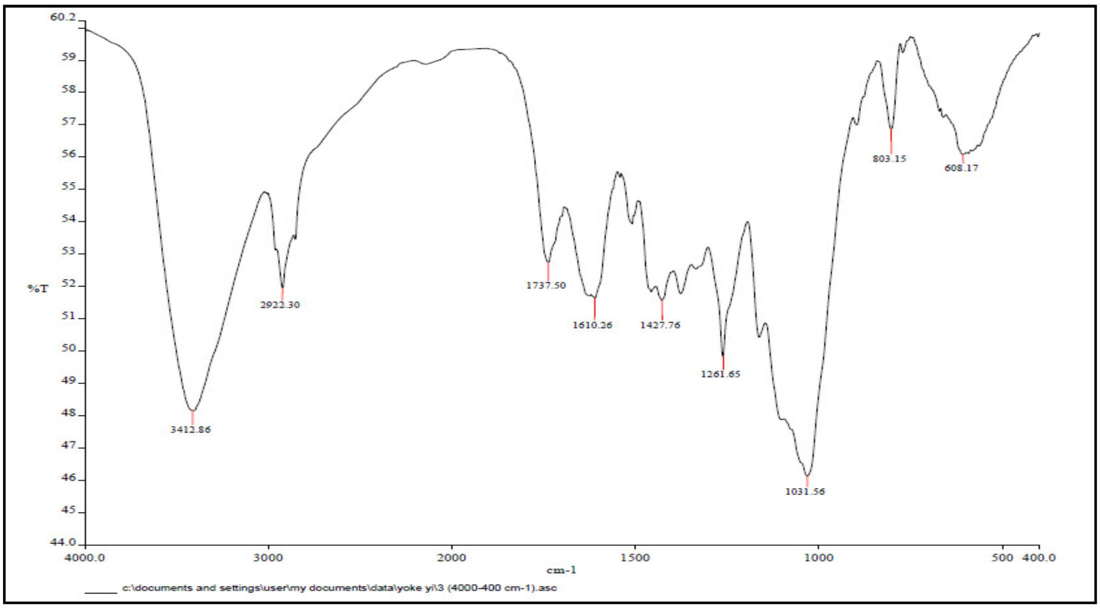

FIGURE 28: FTIR spectra of biocomposite produced with TPS pretreated with EmimAc.

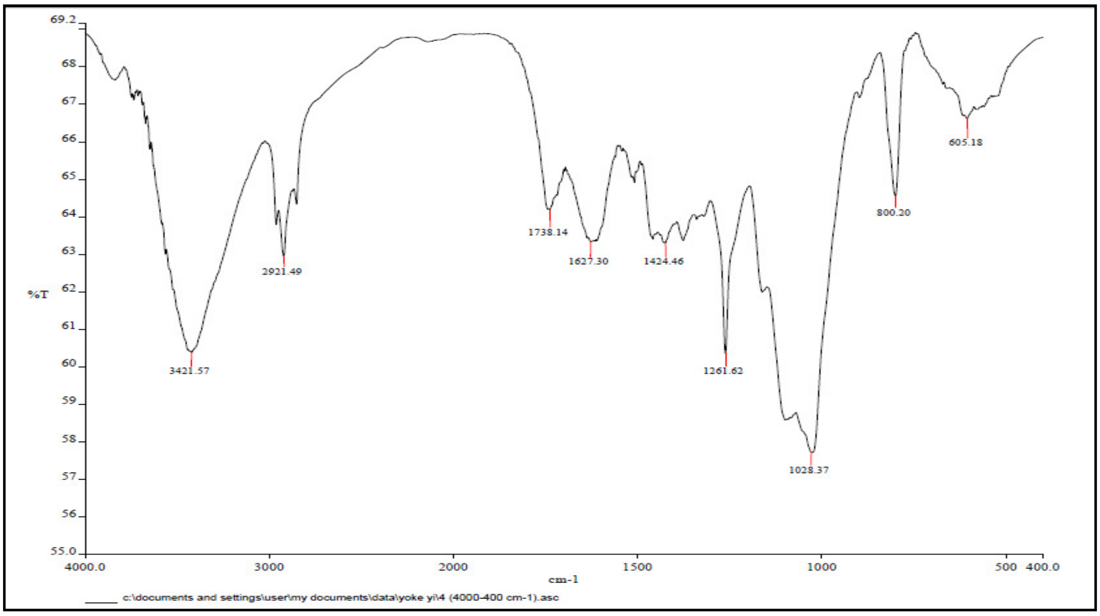

FIGURE 29: FTIR spectra of biocomposite produced with TPS without pretreatment with IL.

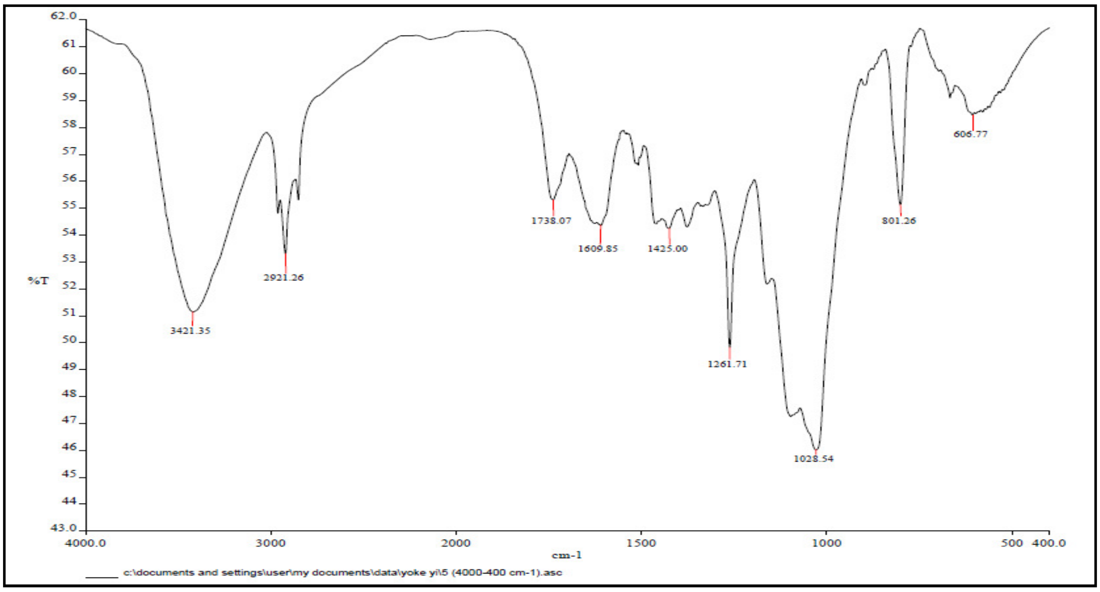

FIGURE 30: FTIR spectra of biocomposites produced with TPS pretreated with BmimTFMS.