STUDY OF CARBON BLACK GENERATED FROM PYROLYSIS OF PASSENGER TYRES ADDED WITH PVOH

KAM WENG JOE

A project report submitted in partial fulfilment of the
requirements for the award of Bachelor of Engineering
(Honours) Mechanical Engineering

Lee Kong Chian Faculty of Engineering and Science
Universiti Tunku Abdul Rahman

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DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

Signature :

Name : KAM WENG JOE

ID No. : 1606962

Date : 12 SEPTEMBER 2020

APPROVAL FOR SUBMISSION

I certify that this project report entitled **"STUDY OF CARBON BLACK GENERATED FROM PYROLYSIS OF PASSENGER TYRES ADDED WITH PVOH"** was prepared by **KAM WENG JOE** has met the required standard for submission in partial fulfilment of the requirements for the award of Bachelor of Engineering (Honours) Mechanical Engineering at Universiti Tunku Abdul Rahman.

Approved by,

Signature :

Supervisor : IR DR BEE SOO TUEEN

Date : 12 SEPTEMBER 2020

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ABSTRACT

Polyvinyl alcohol (PVOH) is a biodegradable and biocompatibility types of biopolymer which can be applied extensively in biomedical fields. However, it is high in cost, lower strength and stiffness when compared with other types of biopolymer. In order to solve these problems, adding reinforcing filler into the PVOH has been extensively attracted the concern of researches. Carbon black (CB) is one of the reinforcing filler which can be used to improve the mechanical properties of PVOH. Furthermore, the carbon black can be obtained through pyrolysis of waste tyres which can enhance the PVOHs' properties. Hence, this work was conducted to investigate the effect of polyvinyl alcohol added with carbon black in terms of mechanical properties, surface morphologies and chemical bonding. The PVOH/CB composites were prepared by using the casting method with 100 phr of PVOH added with different loading level of carbon black particles. The increasing of carbon black up to 10 phr of PVOH/CB composites has increased the tensile strength and Young's modulus of the composite. Conversely, further increment of carbon black up to 30 phr has decreased the mechanical properties. The surface morphologies were observed to have more tearing effect, presence of cavity and higher wavy effect at high loading level of 30 phr of carbon black. Furthermore, the chemical bonding interaction analysis indicated that wavenumber of both O-H and C-H stretching had decreased as the loading level of carbon black increased. The O-H stretching was found to be decreased at higher amount of carbon black (>10 phr) due to the agglomeration effect of carbon black particles, which alter the interaction and bonding between PVOH and CB.

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LIST OF SYMBOLS / ABBREVIATIONS

ASTM	American Society for Testing and Materials
CB	Carbon Black
CCB	Commercial Carbon Black
CS	Chitosan
EPDM	Ethylene Propylene Diene Monomer
FTIR	Fourier Transform Infrared
HAP	Hydroxyapatite
HNT	Halloysite nanotubes
MMT	Montmorillonite
NBR	Nitrile rubber
PANI	poly-aniline
PCB	Pyrolysis Carbon Black
PDI	Polydispersity Index
phr	part per hundred resins
PLA	Polylactic acid
PP	polypropylene
PTFE	Polytetrafluoroethylene
PVAc	poly-vinyl acetate
PVOH	Polyvinyl Alcohol
rpm	revolutions per minute
SEM	Scanning Electron Microscopy
TEB	Tensile energy to break

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CHAPTER 1

INTRODUCTION

1.1 General Introduction

Plastics contamination is as of now one of the most significant natural issues on the planet. More than 320 million tons of plastics are created each year. Removal of utilized tires is a worldwide issue as over half are disposed of with no treatment. It is estimated that the number of used tyres would arrive at 1.2 billion tires yearly till year 2030 (Thomas, Gupta and Panicker, 2016). They comprise an issue because of their huge volume and may have an ecological effect as certain waste products are not environmental friendly and non-biodegradable (Liu, et al., 2016).

The chemical composition of waste tyres usually consists of 22 % of carbon black (CB), 17 % of metals, 6 % of additives (Gómez-Hernández, Panecatl-Bernal and Méndez-Rojas, 2019). Consequently, scientists came out several methods to fabricate polymeric materials from common biomass products (Avérous, 2008). Pyrolysis carbon black (PCB) can be produced through the synthesis of waste tyres as it is one of cost-effective and economical way and able to produce higher value of tyres (Shah, et al., 2006). Thus, the application of reusing of waste plastics is profoundly attractive and beneficially affects the nature and economy.

In order to solve the disposal issues, biopolymers are to be studied to improve their chemical and physical properties to reuse the waste products. The advancement of bio-nanocomposites improves the properties of biopolymer (Yao, 2010). Polymeric nanocomposites have been broadly utilized inferable from their improved properties when contrasted with unadulterated polymers (Han and Fina, 2011). These improved properties can be accomplished with the utilization of incredibly low measure of nanofiller (Chavan, 2015).

1.2 Importance of study

There were few studies or researches done by researchers these few years about the addition of nano-particles to enhance mechanical properties of PVOH matrix for further application. However, there are none of the research had been done by adding carbon black partcles into the PVOH matrix. Thus, the investigation study of composition of carbon black is the most important parameter, which will alter the physical and chemical properties of PVOH matrix, either improve or reduce the mechanical properties, surface morphologies, crystallinity and chemical bonding of polymer composite.

1.3 Problem Statement

Polyvinyl alcohol (PVOH) is a biodegradable polymer utilized in many applications, for example, papermaking, coating applications or in biomedical field due to its biodegradability and biocompatibility properties. However, the commercial cost of polyvinyl liquor is extremely high. This can be done by implementing the PCB from waste tyres to the polyvinyl alcohol matrix to improve the properties of the physical and chemical properties of polyvinyl alcohol. Therefore, the following problem statements of this study were investigated:

1. What are the effects of PVOH/CB blends in terms of mechanical properties and chemical properties?
2. What the effects of PVOH/CB blends in terms of physical properties and the chemical bonding of blends?

1.4 Aim and Objectives

The study was conducted the interaction effect between polyvinyl alcohol and carbon black by evaluating the characteristics tests of the PVOH/CB blends and obtaining the optimum composition of polyvinyl alcohol and carbon black in the blends. In order to achieve the main objectives, sub-objectives were identified as listed below:

1. To investigate the effect of increasing carbon black loading level on the mechanical and chemical properties of PVOH/CB blends
2. To investigate the effect of increasing carbon black loading level on the physical properties and microstructure of the PVOH/CB blends.

1.5 Scope and Limitation of the Study

This study mainly focused on the preparation and characterization of PVOH/CB blends. Blends of polyvinyl alcohol and carbon black were prepared at different composition. The samples were compounded to specimen with the use of casting method. Several tests were carried out to characterize the PVOH/CB blends.

1.5.1 Mechanical Properties

Tensile test was conducted to measure elongation at break, tensile strength, and Young's modulus of PVOH/CB blends. The tensile test was carried out in accordance to ASTM D882 standard to determine properties such as Poisson's ratio and Young's modulus.

1.5.2 Physical Properties and Morphologies

The chemical composition and external morphologies of PVOH/CB blends were analysed by using scanning electron microscope (SEM). The SEM analysis was conducted by scanning the sample with electron beam which produces a two-dimensional image.

1.5.3 Chemical Interactions and Bonding

Fourier transform infrared (FTIR) was used to identify chemical features and shifts that occur because of the interactions between functional groups of the blended specimen. The samples were scanned with infrared light to determine the chemical bonding in PVOH/CB blends.

1.6 Contribution of the Study

The study of this PVOH/CB composite was aimed to apply in biomedical fields, more specifically is used as bone support material. Moreover, the carbon black added is able to enhance the mechanical properties of PVOH as well as improve the bonding interaction. Hence, the improvement of mechanical properties is able to make the polymer composite strong enough.

1.7 Outline of the Report

In this study, there are 5 chapters included as following:

Chapter 1 outlines the background, problem statement, aim and objectives, scope of the study, importance of study, contribution of study and outline of report.

Chapter 2 was focused on literature review of carbon black matrix added with different types of nano-particles and its effect on physical and chemical properties. The contents include the carbon black details, PVOH details, tensile testing, surface morphologies analysis, chemical bonding and interactions analysis.

Chapter 3 mainly present the methodology of this study, which include the materials details, formulation of PVOH/CB composites, sample preparation and characterization testing.

Chapter 4 discuss the results of all the characterization tests, such as mechanical properties through tensile machine, surface morphologies by using SEM and chemical bonding and interaction analysis through FTIR.

Chapter 5 concludes the results discussed and provide recommendations for improvement ofthis study.

CHAPTER 2

LITERATURE REVIEW

2.1 Carbon black (CB)

2.1.1 Properties of Carbon Black

Carbon black is a finely divided carbon particulate produced by thermal decomposition which consists of spherical sizes of diameter 10 nm to 100 nm (Marsh and Rodríguez-Reinoso, 2006). The carbon black is chemically composed of carbon content with more than 98 % but oxygen can be formed due to hydroxyl and carboxyl group located on the surface. The basic properties of it are determined by the composition of functional groups that contains oxygen.

It is water soluble and has acidic characteristics with a surface-to-volume ratio of 50 nm. According to (Gómez-Hernández, Panecatl-Bernal and Méndez-Rojas, 2019), an experiment on the water suspension of carbon black particulate was conducted and found that it has acidic pH value of 5. The particle size distribution of carbon black (CB) has a large pore size dispersion with a polydispersity index (PDI) value of 0.273 due to the particles are chemically bonded with long and large chain agglomerations (Gómez-Hernández, Panecatl-Bernal and Méndez-Rojas, 2019). The bond between the large agglomerates is weak and can be destroyed easily due to the weak interactions of Van der Waals forces.

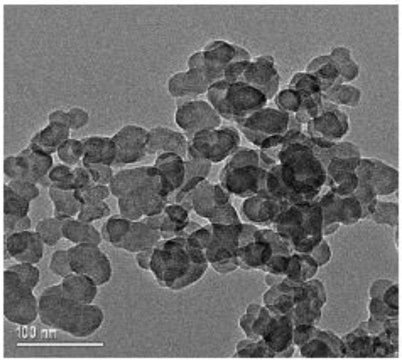


Figure 2.1: SEM micrograph of carbon black (Marsh and Rodríguez-Reinoso, 2006).

2.1.2 Applications of Carbon Black

Carbon black was extensively used as reinforcement filler in sensors, detectors and coating application packaging because it is able to enhance the mechanical properties of the polymer composites (Li, et al., 2003; Wu, et al., 2009). On the other hand, it can also be used as electromagnetic shielding and supercapacitor due to its conductive properties (Zhou, et al., 2016). It was widely applied in the electronic industry due to its low cost and having superior electrode properties for supercapacitors.

2.2 Polyvinyl Alcohol (PVOH)

2.2.1 Properties of Polyvinyl Alcohol

Polyvinyl alcohol (PVOH) is a water soluble polymer material with high crystallinity structure and good durability, biodegradability and chemical resistance (Sin, et al., 2010). The properties of the PVOH can be determined by the crystallinity, polymerization conditions and the distribution of hydroxyl groups (Chiellini, Corti and Solaro, 1999).

It exhibits high crystallinity due to the high concentration of hydroxyl groups located on the monomer residuals without disrupting the lattice structure (Gohil, Bhattacharya and Ray, 2006). The solubility of the water soluble synthetic polymer depends on the degree of crystalline structure and its polymerization conditions (Hassan and Peppas, 2000). The higher the degree of hydrolysis will result in a lower solubility and higher difficulty to crystallize. PVOH is an atactic polymer with high biocompatibility that can be produced through the hydrolysis of polyvinyl acetate. Thus, it is very compatible to blend with other biopolymers to improve the physiochemical properties of the polymer blend due to its overall good hydrophilic properties and high polarity (Bee, et al., 2018).

2.2.2 Synthesis of Polyvinyl Alcohol

PVOH is unable to directly produce through vinyl alcohol polymerization, thus it is usually produced from polyvinyl acetate (PVAc) through hydrolysis process in alcohol solution (Rowe, Sheskey and Cook, 2009). This hydrolysis process can be carried out through suspension polymerisation or free radical mechanism. Acetate groups will be removed out from the PVAc due to the presence of hydroxyl group which come from the alcohol solution added (Illanes, 2010). The mechanism of the hydrolysis reaction was shown in Figure 2.2. Furthermore, the PVOH produced has the same degree of polymerisation with the PVAc (Ogur, 2005).

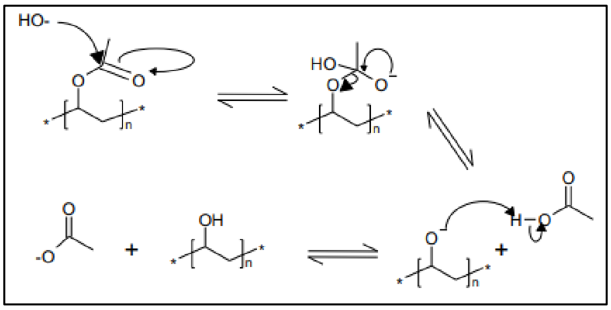


Figure 2.2: Mechanism Flow of Hydrolysis of PVOH from PVAc (Illanes, 2010).

2.2.3 Analysis of Polyvinyl Alcohol composites

(Ashraf, Zebarjad and Hadianfard, 2019) were studied a polymeric foam of polyvinyl alcohol with the addition of hydroxyapatite nanoparticle (PVOH/HAP film). They were found that addition of HAP with the PVOH will tend to improve the water absorbance with a range of 58 % to 71 %. Besides, it also can improve the bioactivity and water uptake. Furthermore, the researchers also found that the biodegradability PVOH-HAP nanocomposite has successfully taken place with body simulated liquid at 37 ˚C.

(Chai, et al., 2009) stated that readily biodegradable plastics can be produced from the polyvinyl alcohol blended with the thermoplastic and water-soluble starch. The thermoplastic starch cannot form a stable film by itself due to its’ sticky texture. Therefore, it can blend with the PVOH to form a film of biodegradable plastic. Besides that, the thermoplastic starch can improve the melt strength and elongation properties of the PVOH. The PVOH-starch film was found to have improvement of moisture absorption and higher resistance to some chemicals.

(Ghaffari-Moghaddam and Eslahi, 2014) studied a type of polymer nanocomposite involved the use of PVOH, which is a polyaniline/polyvinyl alcohol/Ag (PANI/PVOH/Ag) nanocomposite. The similarities of both PANI and PVOH are highly biocompatible and biodegradable polymers. However, PANI is generally insoluble in water, thus PVOH was used with a unique purpose of improving the solubility of PANI. The researchers were found that this nanocomposite polymer has a very good efficiency in nanoparticle coating due to its relatively small particle size, which is about 10 nm to 17 nm. Besides, an antibacterial study was also carried out in this work, unfortunately, the results showing that it is far from the expectation. However, it is found effective against the two bacteria used in this work, such as Staphylococcus aureus and Escherichia coli.

(Bourakadi, et al., 2019) implemented research on chitosan/ polyvinyl alcohol/modified thiabendazolium-montmorillonite (CS-PVOH-MMT) biopolymer nanocomposite. The Young’s modulus and tensile strength were increased by adding MMT with the CS/PVOH. Besides that, the presence of the thiabendazolium compound affected the antimicrobial activity of the new elaborate films in a positive way. They were found that the CS-PVOH-MMT films have performed a good activity of antimicrobial, which against all the bacterium used in this testing, such as aeruginosa, S. aureus and E. coli.

(Gaaz, et al., 2015) conducted research of polyvinyl alcohol-halloysite nanotubes nanocomposite (PVOH-HNTs). The HNTs have excellent mechanical property. The addition of 5 wt% of HNTs in the PVOH-HNTs nanocomposite can help to increase 300 % of the tensile strength, which can triumph over the relatively low strength of PVOH. Besides that, the low thermal stability of PVOH can also be solved by the addition of HNTs due to its’ excellent stability even at high temperature. These PVOH-HNTs blends tend to increase the application in biomedical fields by not concerned about the low strength and thermal stability of PVOH.

2.2.4 Applications of Polyvinyl Alcohol

Polyvinyl alcohol (PVOH) is widely used as paper coatings and sizing in the pulp and paper industry, hydrophilic coatings in the biomedical industry and food packaging in the food industry (Chiellini, Corti and Solaro, 1999). The main usage of the synthetic polymer is to function as a stabilizer of dispersion systems due to its non-ionic surface active agent (Arvanitoyannis, et al., 1997).

2.3 Pyrolysis

2.3.1 Background of Pyrolysis

The decomposition of biomass when heated with temperature of 500 ˚C to 800 ˚C in an inert atmosphere is known as pyrolysis (Jahirul, et al., 2012). It is a waste valorization approach that transforms waste products into gas, liquid, and solid materials.

The gas, liquid, and solid materials of the by-products of organic matter are usually known as bio-syngas, bio-oil and bio-char respectively. Higher quality of pyrolytic residue can be obtained by using heterogeneous catalyst in the thermal decomposition to further improve the chemical properties of the pyrolytic products (Iliopoulou, Triantafyllidis and Lappas, 2019).

2.4 Tensile Test

2.4.1 Background of Tensile Testing Machine

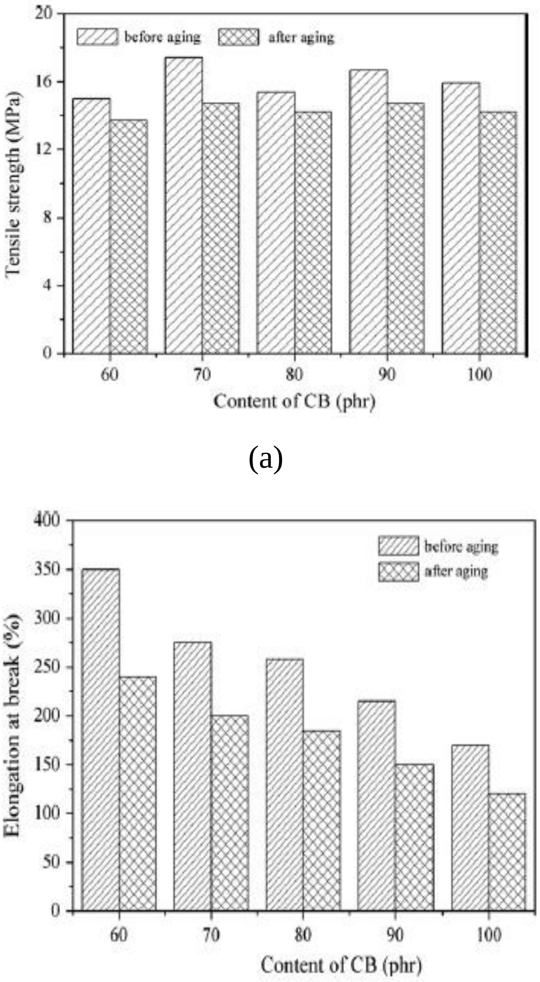
Polymer films' tensile test should undergo bending and slip by using the methods found in American Society for Testing and Materials (ASTM) D882. The tensile strength, yield, modulus and elongation are tested by pulling the polymer film until it breaks. The samples used for ASTM D882 can be prepared in a rectangular shape with about 1 mm thick instead of dumbbell shape. The tensile elastic modulus is also known as the stiffness of the polymer films. The toughness of the sample is determined by the tensile energy to break (TEB). TEB is the overall energy absorbed up to the point of breaking per volume of the sample.

A few trial runs of testing should be carried out for one set of same composition samples by using ASTM D882 to get an average result of the samples. Firstly, place the sample in the grips of machine and make sure the sample is aligned perpendicularly with grips. Next, tighten the grips with the polymer films evenly and firmly to prevent the slipping occur during the testing. The next step is just to start the machine and wait until it breaks. The integration of the stress-strain curve is then performed by the software (TestResources, 2019).

2.4.2 Tensile Test of CB polymer composites

(Jovanović, et al., 2013) had studied nitrile rubber/ethylene propylene diene monomer (NBR/EPDM) rubber blends with added carbon black particles. The results of the tensile strength and tensile extension at maximum tensile stress on NBR/EPDM rubber blends with various amount of carbon black have been determined in his study. The tensile strength was shown an increased value when increased the amount of carbon black to 70 phr which was shown in Figure 2.3 (a). However, by increasing the amount of carbon black, it was shown a decrease of tensile strength as amount of CB increased. This is because the higher amount of carbon black could weaken the interaction effect between the NBR and EPDM blends. The carbon black will agglomerate and formed a larger particles size, which will reduce the interfacial adhesion effect of the composite polymer. Furthermore, the elongation at break were decreased when the amount of carbon black are increased. Figure 2.3 (b) showed the results of elongation at break with different amount of carbon black. This is because the carbon black will reduced the interaction of NBR and EPDM, hence the chain sliding ability decreased and tends to decrease the elongation at break.

(Kanbur and Küçükyavuz, 2009) studied a research on the interactive of polypropylene (PP) and carbon black reinforcement polymer composite system. The samples were then prepared by using injection molding and compression molding for comparison of mechanical properties. The results on the mechanical properties are shown in Figure 2.4 (a) tensile strength, (b) Young’s modulus and (c) elongation at break. As shown in Figure 2.4 (a), there is an increased of tensile strength when the carbon black raised from 1 phr to 30 phr by using the injection molding. This is because the presence of higher amount of carbon black might increase the effective local reinforcing points and increase in specific surface area of carbon black in the PP blends. This will improve the binding of carbon blacks with PP by the Van der Waals force. Furthermore, Young’s modulus is increased when the amount of carbon black increased in the both molding method. This is because the fillers are promoting a strong reinforcing action and it will also induce higher polymer blends rigidity. Furthermore, the elongation at break was shown a decreased value with increasing amount of carbon black. Figure 2.4 (c) showed the results of elongation at break with different amount of carbon black. The is due to the carbon black could enhance the interaction of PP, hence the chain sliding ability decreased and tends to decrease the elongation at break.



(a)

(b)

Figure 2.3: Results of (a) Tensile strength (b) Elongation at Break of NBR/EPDM composite at different loading level of carbon black (Jovanović, et al., 2013).

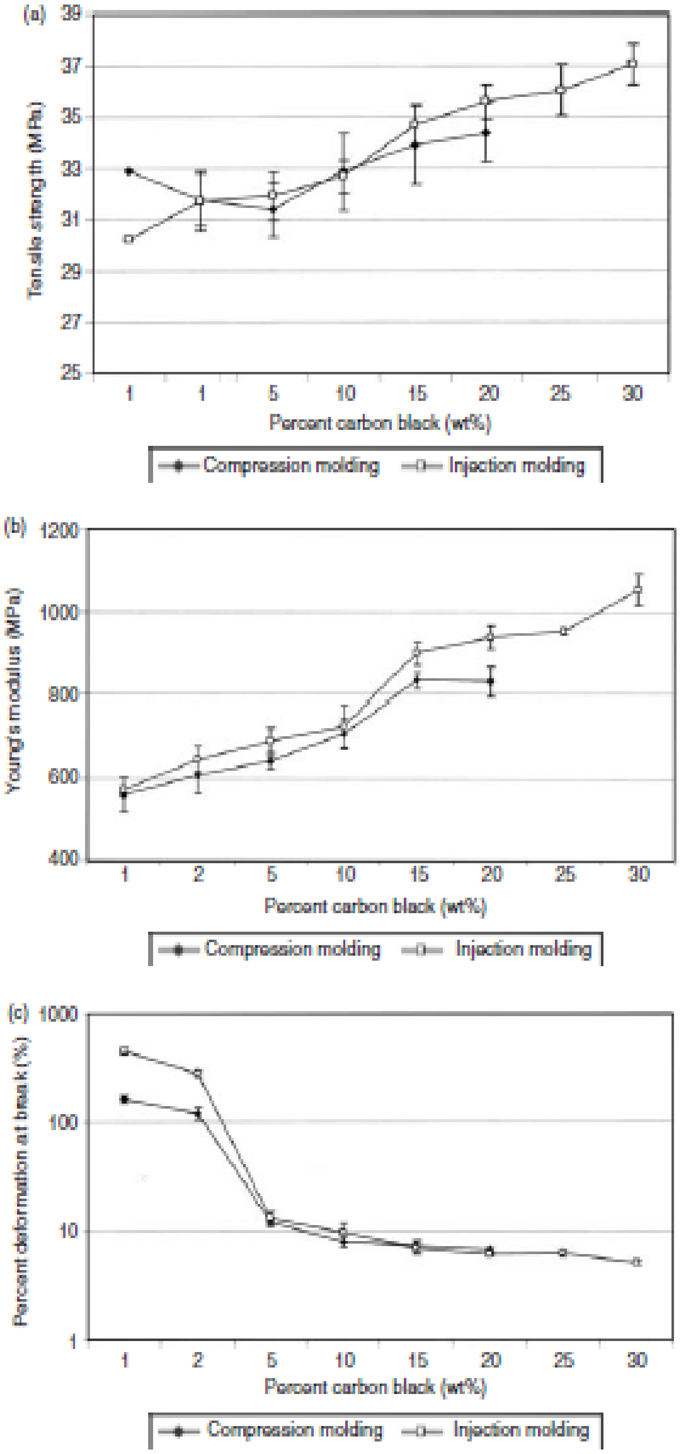


Figure 2.4: Results of (a) Tensile Strength, (b) Young's Modulus and (c) Elongation at break of PP nanocomposites at different loading level of carbon black (Kanbur and Küçükyavuz, 2009).

2.5 Scanning Electron Microscopy (SEM)

2.5.1 Background of SEM

SEM analysis is applied to determine the surface morphology, chemical composition and orientation of specimens. The electrons are used in the SEM to form an image of the surface of the samples. The magnification of 1000X can be achieved by the modern light microscopes. However, SEM can reach more than 30 000X of magnification.

The electron gun is initially used to generate the electrons, which act as a cathode in this stage. Meanwhile, the anode is being propelled by these electrons due to strong electric fields. The electrons are then condensed through the condenser lens and enter into the objective lens. The schematic of the SEM is shown in Figure 2.5. The primary electron is then struck to the conductive sample to a depth which depends on the electron energy level. Then, the secondary electron and backscattered electrons will strike the sample and reflect out from the sample. The secondary electron and backscattered detectors are used to measure the reflected electrons. Finally, the image will be performed on the screen (Oatley, Nixon and Pease, 1966).

Furthermore, the procedure for handling the SEM is needed to be considered. The sample is initially placed on a sample stub and then place into the gold sputtering system. The layer of gold thickness used depends on the geometry of the sample. Besides, a longer sputtering time is needed for the more rough or porous surface. Next, the stub is removed from gold sputtering system. The sample is then inserted into the sample chamber. Pump is then turned on to make the environment inside the chamber become a vacuum. Next, select the operating voltage from the range of 1 kV to 30 kV. Adjusting the focus until reached the desired magnification to get the desired image. The last step is just saving and rename the images that were done by the SEM (Sweney, Laible and Shapiro, 1976).

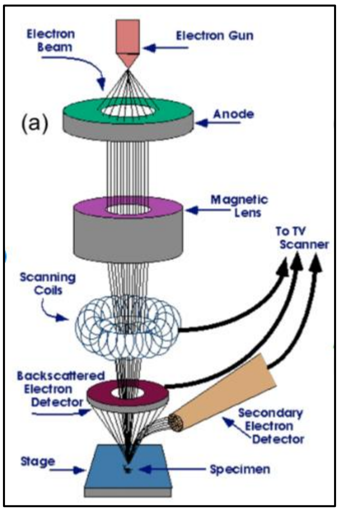
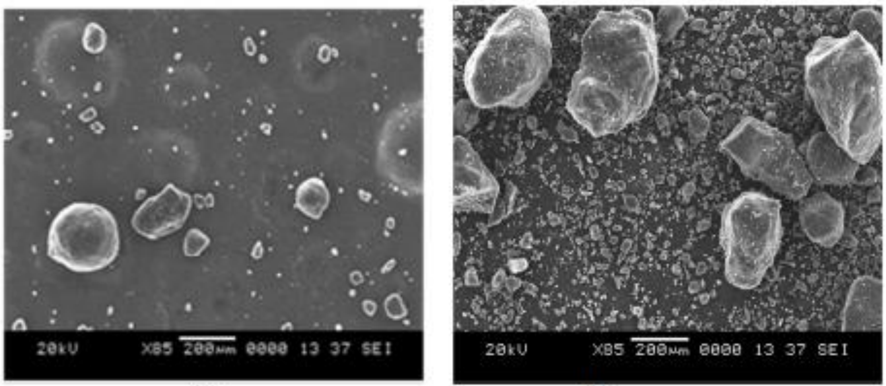


Figure 2.5: Schematic of SEM (Goldstein, et al., 2003).

2.5.2 SEM Analysis of Pyrolysis Carbon Black (PCB) and Commercial Carbon black (CCB)

(Martínez, et al., 2019) had conducted SEM characterization test to determine the surface morphological characteristics of PCB and CCB. In Figure 2.6, it was shown that the commercial carbon black had a more homogenous, fine and smooth grain structure than the PCB.

The low structure of PCB is due to the usage of different CB grades in the chemical composition of the tyres. Structure is used to evaluate the bulkiness and size of the agglomerates. It was found that the enhanced properties of CB depend on the structure of CB as lower structure of CB has low degree of aggregation which is not easily disperse.



(a) (b)

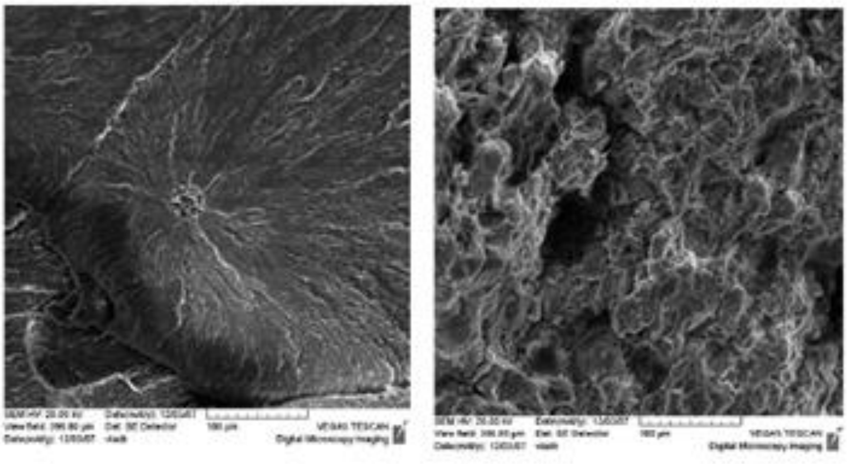
Figure 2.6: SEM analysis of (a) CCB, (b) PCB (Umi Fazara, et al., 2013).

2.5.3 SEM Analysis of CB polymer composites

(Jovanović, et al., 2013) found that the dispersion of NBR/EPDM rubber composite with 70 phr of carbon black were not homogenous in the blends as shown in Figure 2.7. This is due to the poor compatibility between NBR/EPDM composite which results in a poor interfacial adhesion effect with carbon black. It can be observed that there are irregular shapes and the surface is not uniform in Figure 2.7.

(Aderikha and Shapovalov, 2010) were also found that by increasing the amount of CB, the mechanical properties of polytetrafluoroethylene/carbon black (PTFE/CB) composite were weaker which shown in Figure 2.8. It was found that a flakes-like structure and agglomerated particles were formed under SEM images, which shown in Figure 2.8 (b). The presence of this structure is due to the dispersion of PTFE and carbon black not homogenous in the PTFE blends. This poor dispersion of carbon black in the PTFE tends to decrease the interfacial adhesion of carbon black with PVOH. Moreover, the agglomeration has clearly occurred in the 10 phr of carbon black added PTFE composites fractured surface. This will reduce the tensile strength of PTFE/CB composite and weaken the reinforcement effect of carbon black. This lower tensile strength of PTFE/CB composite might have higher brittle properties, which can easier break or crack and might change in shape.

Figure 2.7: SEM Micrograph of NBR/EPDM rubber composite with 70 phr of carbon black (Jovanović, et al., 2013).



(a) (b)

Figure 2.8: SEM Micrograph of PTFE composite added with (a) 1 phr of carbon black (b) 10 phr of carbon black (Aderikha and Shapovalov, 2010).

2.6 Fourier Transform Infrared Spectroscopy (FTIR)

2.6.1 Background of FTIR

FTIR analysis is applied for determination of chemical bonding and interactions present in the samples. FTIR consists of a light source, interferometer, sample compartment, reflected light detector, amplifier and converter. The light source is initially reflected and pass through the beam splitter in the interferometer. The Michelson interferometer is normally used in FTIR. It consists of a beam splitter, a stationary and a movable perpendicular mirrors. The reflected light from the beam splitter is then pass through the sample in the sample compartment and reflect to the detector eventually. The signal is then converted by the amplifier to the digital signal and the converter will convert the results and perform the Fourier transform spectrum (Adam May and Mike Ellicott, 2019). The schematic of the FTIR is shown in Figure 2.9. Besides that, the frequency range can be measured by FTIR is from 4000 cm^{-1} to 600 cm^{-1}.

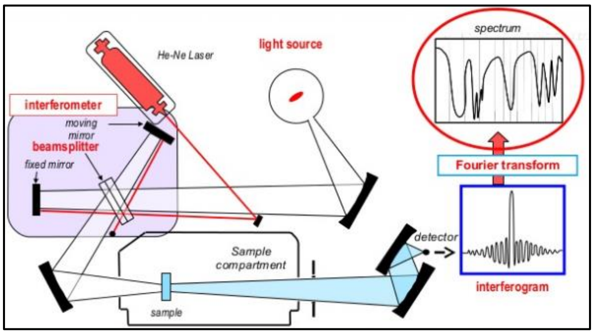


Figure 2.9: Schematic diagram of FTIR (PerkinElmer, 2009).

2.6.2 FTIR Analysis of CB polymer composites

(Wen, et al., 2012) carried out FTIR test on the PP blends with different amount of carbon black and the results were shown in Figure 2.10. It was reported that there are peaks at 1240 cm^{-1} and 886 cm^{-1} found in PP/CB composites with 2.5 phr and 5 phr of carbon black respectively as shown in Figure 2.10. The wave number of the stretch is found to have a decrement as carbon black increased from 2.5 phr to 5 phr. The wave number at 886 cm^{-1} indicated the presence of carbonate compound whereas wave number at 1240 cm^{-1} indicated the presence of CH blends. This indicates the formation of new chemical bonds as it does not belong to the vibration modes of PP.

(Wang, et al., 2008) also carried out the FTIR spectrum in their research with polylactic acid (PLA) blends with different amount of carbon black and the results were shown in Figure 2.11. The wavenumber at 1749 cm^{-1} indicated the presence of stretching of carbonyl groups in PLA blends; wave number range from 1182 cm^{-1} to 1043 cm^{-1} indicated the presence of phosphate compound. Moreover, the wave number is decreased as the amount of carbon black increased. This is due to the agglomeration of carbon black occurred when a large amount of carbon black presence in PLA, which reduces the dispersion of carbon black. This will increase the H-bond formed inside the PLA.

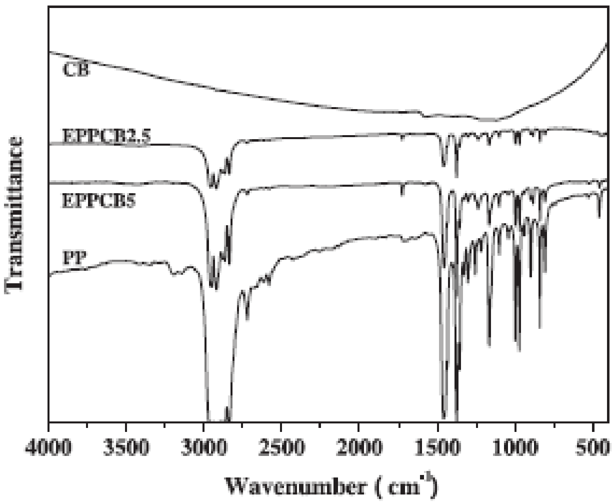


Figure 2.10: FTIR spectrum of PP composites with different loading level of CB contents (Wen, et al., 2012).

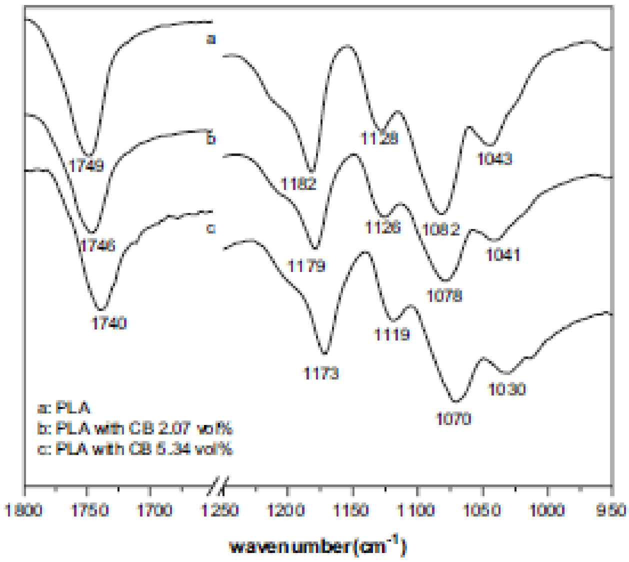


Figure 2.11: FTIR spectrum of PLA composites with different loading level of CB contents (Wang, et al., 2008).

2.7 Summary

In the literature review, the addition of CB into different types of nano-particles will give different effects to the polymer composites. In short, the tensile strength, Young’s modulus and tensile extension at maximum tensile stress had been induced by adding carbon black. If the loading level of carbon black added is too much, it could reduce the tensile properties. For the surface morphologies, the structure of polymer composite with absence of agglomerated particles, lesser brittle structure, lesser tearing effects with separated phase and higher continuity and smooth surface are illustrated as a better dispersion of nano-particles in polymer composite. For chemical bonding and interaction of CB polymer composites, most of the researches found that the increasing amount of carbon black led to a lower wave number in the FTIR spectrum.

CHAPTER 3

METHODOLOGY AND WORK PLAN

3.1 Materials

Fully hydrolysed PVOH with a grade of Denka Poval, K-17C was manufactured by Denki Kagaku Kougyo Kabushiki Kaisya (DENKI) Company. The hydrolysis and viscosity of this PVOH are from 87 mol% to 89 mol% and from 45 mPa·s to 55 mPa·s. Next, carbon black was obtained by derivation of waste tyres via pyrolysis process.

3.2 Formulation

The formulations for the casting samples were shown in Table 3.1. The content of PVOH for all samples was set at constant value of 100 phr, while the amount of carbon black (CB) was increased accordingly as shown in Table 3.1. All the samples were given an abbreviation to easily identify different sets of samples.

Table 3.1: Composition of CB-PVOH in Casting Samples.

Sample	PVOH (phr)	CB (phr)
P-110	100	10
P-115	100	15
P-120	100	20
P-125	100	25
P-130	100	30

3.3 Sample Preparation

The PVOH/CB samples were prepared using the solution casting method. Firstly, the hotplate was used to heat the 250 ml distilled water in a 500 ml beaker to a temperature of 97 ˚C with a tolerance of 2 ˚C until it reached the boiling point. Next, 10 g of PVOH resin and 50 ml of distilled water were added into a 250 ml beaker and were placed into the pre-heated 500 ml beaker. After that, a stirrer with constant rotating velocity of 350 rpm was functioned to agitate the mixture of water and polyvinyl alcohol until the PVOH resin was totally diffused in the distilled water. Then, various amounts of CB were added into PVOH solution as shown in Table 3.1 and agitation was carried out again with the same condition. The mixture was kept stirring with the constant speed and temperature until the homogenous mixture was formed. Later, the mixture solution was casted onto the diameter petri dishes and the casted samples were dried inside the oven at 65 ˚C. The samples were then removed from the petri dishes and stored in a sealed plastic bag for storage purpose.

3.4 Characterization test for PVOH/CB composites

3.4.1 Tensile Test

Tensile test was conducted using Shimadzu EHF-EM050K1-020-0A Tensile Machine. The samples were cut into smaller sizes with accordance to standard ASTM D882. The samples were cut into rectangular size with a width of 0.5 cm by length of 7 cm. The tensile machine was used to record the Young's modulus and tensile strength of the samples by using the Bluehill software.

Figure 3.1: Shimadzu EHF-EM050K1-020-0A for Investigation of Tensile Properties of PVOH/CB composites.

3.4.2 Scanning Electron Microscopy (SEM)

The surface morphology analysis was carried out with the use of Hitachi S-3400N SEM. The samples were cut into smaller sizes with length of 3 cm which are to be placed on the copper stud with the position of the fractured surface facing upward. The samples were then covered with palladium and gold with the use of EMITECH SC7260 Sputter Coater to be scanned with 15 kV of electron beam.

3.4.3 Fourier Transform Infrared (FTIR)

The chemical bonding of the PVOH/CB films was determined by using the ThermoScientific Nicolet iS10 Fourier Transform Infrared Spectroscopy with wavelength range from 4000 cm^{-1} to 400 cm^{-1}. The samples were placed on the sample holder before scanning.

Figure 3.2: ThermoScientific Nicolet iS10 Fourier Transform Infrared Spectroscopy Used for Investigation of the Chemical Interaction and Bonding of PVOH/CB composites.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Mechanical Properties Analysis

4.1.1 Tensile Test for PVOH/Carbon Black composites

4.1.1.1 Tensile Strength

The tensile strength of PVOH/CB composites with increasing the amount of CB from 10 phr to 30 phr were shown in Figure 4.2 and Table A-1.

By referring to Figure 4.1, the tensile strength of the PVOH composites decreased significantly when the carbon black was increased gradually up to 15 phr. The tensile strength was observed to decrease slightly when the carbon black loading level was increased to 20 phr. The tensile strength of PVOH/CB composites reached the highest value of 99.24 MPa when added with 10 phr of carbon black. The tensile strength has decreased from 99.24 MPa to 74.17 MPa from 10 phr to 20 phr of carbon black. The reduced tensile strength was attributed due to the presence of poor dispersion and also the weak interaction between the PVOH matrix and the carbon black particles (Jovanović, et al., 2013). Nevertheless, a small amount of carbon black (≤ 10 phr) could provide a homogeneous distribution of carbon black on PVOH matrix and further improved the hydrogen bonding formed by the O-H functional group of the PVOH matrix. The hydrogen bonding then allows the carbon black particles to bind strongly with the PVOH matrix and improved its mechanical properties.

Besides, it was also found that the tensile strength increased from 74.17 MPa to 86.63 MPa, and then decreased again to 67.11 MPa when added with 30 phr of carbon black. The results indicated that tensile strength of PVOH/CB decreased with increasing amount of carbon black. This is due to the PVOH/CB composites are not well compatible with each other as improper dispersion of carbon black in PVOH occurred which leads to the agglomeration of the carbon black particles. The agglomerated carbon black particles eventually reduced the bonding interactions with PVOH matrix and weaken the tensile strength of the PVOH/CB composites (Jovanović, et al., 2013). Furthermore, the agglomerated particles also acted as the stress concentration point when subjected to extension where the stress is unable to be transferred effectively throughout the PVOH matrix (Bee, et al., 2018). This indicates that the carbon black was considered to give less reinforcing effect to PVOH matrix.

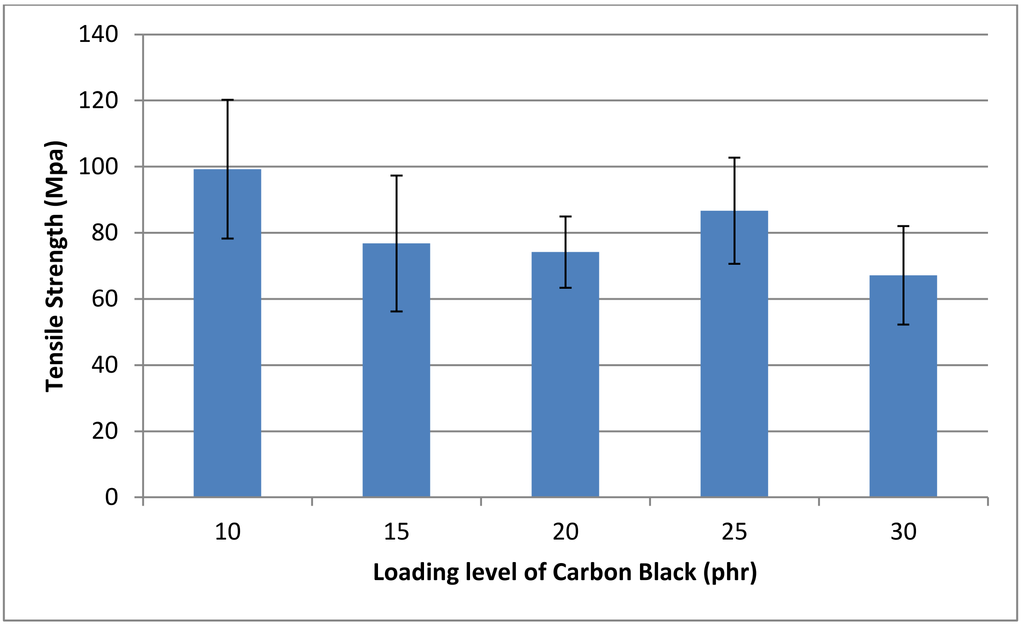


Figure 4.1: Tensile strength of PVOH composites with different loading level of carbon black.

4.1.1.2 Young’s Modulus

Figure 4.2 and Table A-1 shows the Young’s modulus of the PVOH/CB composites when different loading level of carbon black was added. The Young’s modulus of the PVOH/CB composites has increased slightly when amount of carbon black increased from 20 phr to 25 phr. The addition of carbon black particles would form hydrogen bond with the hydroxyl functional group of PVOH chains within the PVOH matrix. The presence of carbon black could also enhance the local stiffening effect by improving the interlocking effect of carbon black and PVOH (Kanbur and Küçükyavuz, 2009). Hence, the stress acted can be transferred throughout the whole PVOH matrix and the rigidity of the PVOH matrix was significantly increased. Therefore, the Young’s modulus increased as the rigidity of PVOH composites increased.

In contrast, the Young’s modulus of PVOH composites started to show a decrement trend as the amount of the carbon black further increased to 30 phr. This observation can be further explained due to the poor interaction and interfacial adhesion performance of carbon black with PVOH at a higher loading level of carbon black. The poor dispersion of carbon black are more likely to agglomerate in the PVOH matrix and larger size and irregular shape of carbon black particles would further reduce local stiffening of carbon black with PVOH. Hence, the agglomerated particles which acted as a stress concentration point will promote crack propagation and this causes the rigidity and Young’s modulus of PVOH/CB composites to decrease (Bee, et al., 2018).

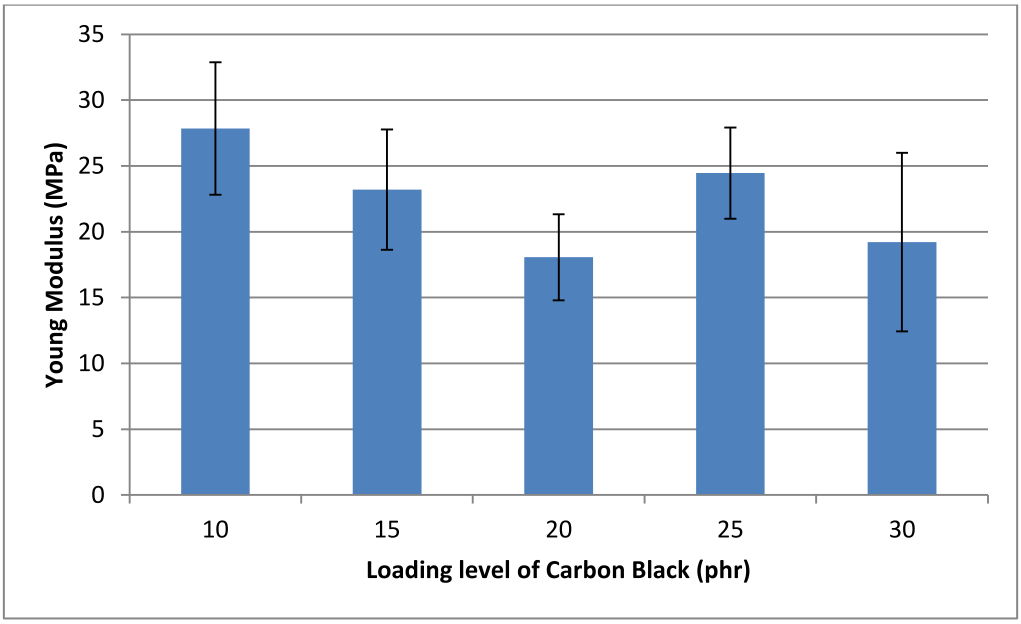


Figure 4.2: Young’s modulus of PVOH composites with different loading level of carbon black.

4.2 Surface Morphologies Observations

4.2.1 Scanning Electron Microscopy (SEM) Analysis for PVOH/Carbon Black composites

The SEM results of PVOH/CB have determined through scanning electron microscopy under a magnification of 3000 times was shown in Figure 4.3.

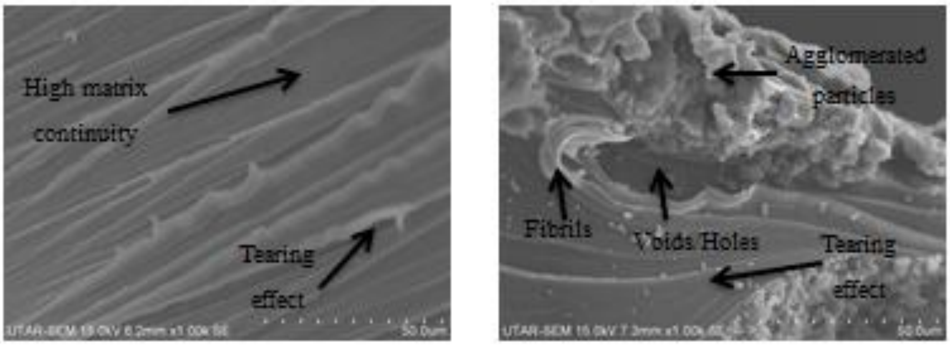
By referring to Figure 4.3 (a), (b), (c) and (d), the tearing effect was present in all surface morphologies of PVOH/CB composites. The outstanding interaction effect of PVOH and carbon black particles have further improved the effectiveness in transferring the applied straining stress from particles to PVOH. The addition of 10 phr of carbon black has progressively reduced the tearing effect of PVOH/CB composite with comparison of PVOH composite with 20 phr of carbon black. This is due to the good interfacial adhesion effect of carbon black particles with PVOH/CB blends, which reduce the numbers of stress concentration points when subjected to strain (Jovanović, et al., 2013). Furthermore, the surface morphologies of PVOH composite with 10 phr of carbon black were also observed to be most continuous when compared with the other PVOH/CB composites. This indicated that the addition of 10 phr of carbon black into the PVOH/CB blends has highly improved the compatibility between PVOH, carbon black in PVOH/CB composites. Besides, the effective interfacial adhesion effect of carbon black with PVOH has also further reduced occurrence of cavities in the PVOH/CB composites. Thus, it enhances the interfacial bonding of PVOH/CB.

Not only that, the formation of fibrils induced on the fractured surface of the polymer composites can be clearly observed in the PVOH/CB added with 20 phr of carbon black. This might due to the presence of carbon black which induced the number of stress concentration point in the PVOH during straining and causing tearing of PVOH. Thus, this further weakened the tensile strength of the PVOH/CB composites. In addition, an extreme wavy effect can be observed in Figure 4.3 (d). This indicated that the 25 phr of carbon black has induced the elongation effect in the PVOH/CB composite. The formation of wavelike structures was caused by the rebounding effect of the PVOH/CB composite when the breakage occurred during elongation (Bee, et al., 2018). This might due to excellent dispersion of carbon black in PVOH/CB blends, which enhanced the interfacial effect of PVOH with carbon black, hence strengthened the PVOH/CB composites. This indicates that wave like structure in PVOH/CB composites resulted in a greater elongation under plastic deformation when subjected to straining.

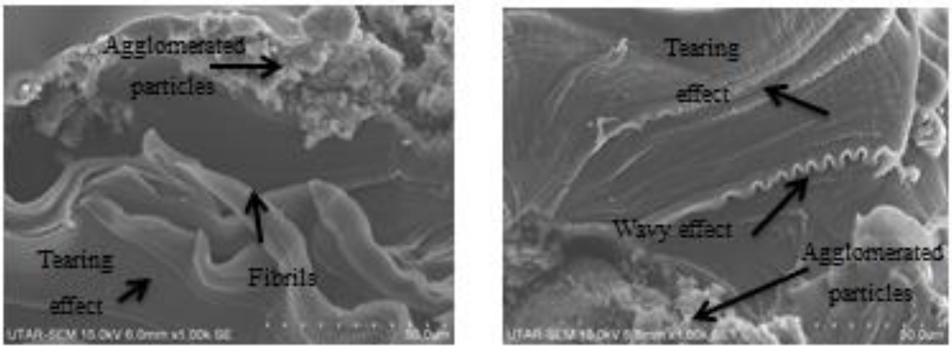
Moreover, the occurrence of cavities also known as voids can be observed on the PVOH/CB composite added with more than 10 phr of carbon black. This might due to the high amount of carbon black were added into the PVOH/CB blends, which might cause a poor interaction of carbon black with

PVOH and led to serve agglomeration effect of carbon black particles in polymer matrix (Aderikha and Shapovalov, 2010). Thus, the PVOH/CB composite added with high amount of carbon black might be easier to be break when subjected to stress. This indicates that the cavities would lead to lower Young’s modulus and tensile strength in the PVOH/CB composites.

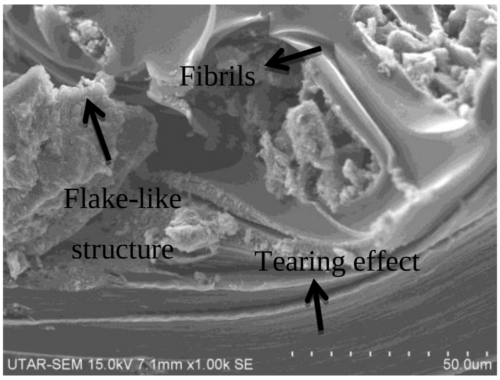
Besides that, the flake like structures can also be observed in the PVOH/CB composites with the addition of 30 phr of carbon black as showed in Figure 4.3 (e). This might be due to the poor dispersion of carbon black particles in the PVOH/CB blends which led to a weaker interfacial adhesion effect and interaction of PVOH/CB composites. The flake like structure was present due to the immediate breaking of the PVOH/CB composite without any elongation effect during the tensile test (Raphey, et al., 2019). This is because the poor dispersion of carbon black tended to cluster together and formed agglomerated carbon black particles, causing the absence of rebounding effect in the PVOH/CB composites. Thus, the flake like structure in PVOH/CB composites results in a lower Young’s modulus.



(a) 10 phr carbon black (b) 15 phr carbon black



(c) 20 phr carbon black (d) 25 phr carbon black



(e) 30 phr carbon black

Figure 4.3: Scanning Electron Microscopy of PVOH composites with different loading level of carbon black at magnification of 1000X.

4.3 Chemical Bonding and Interaction Analysis

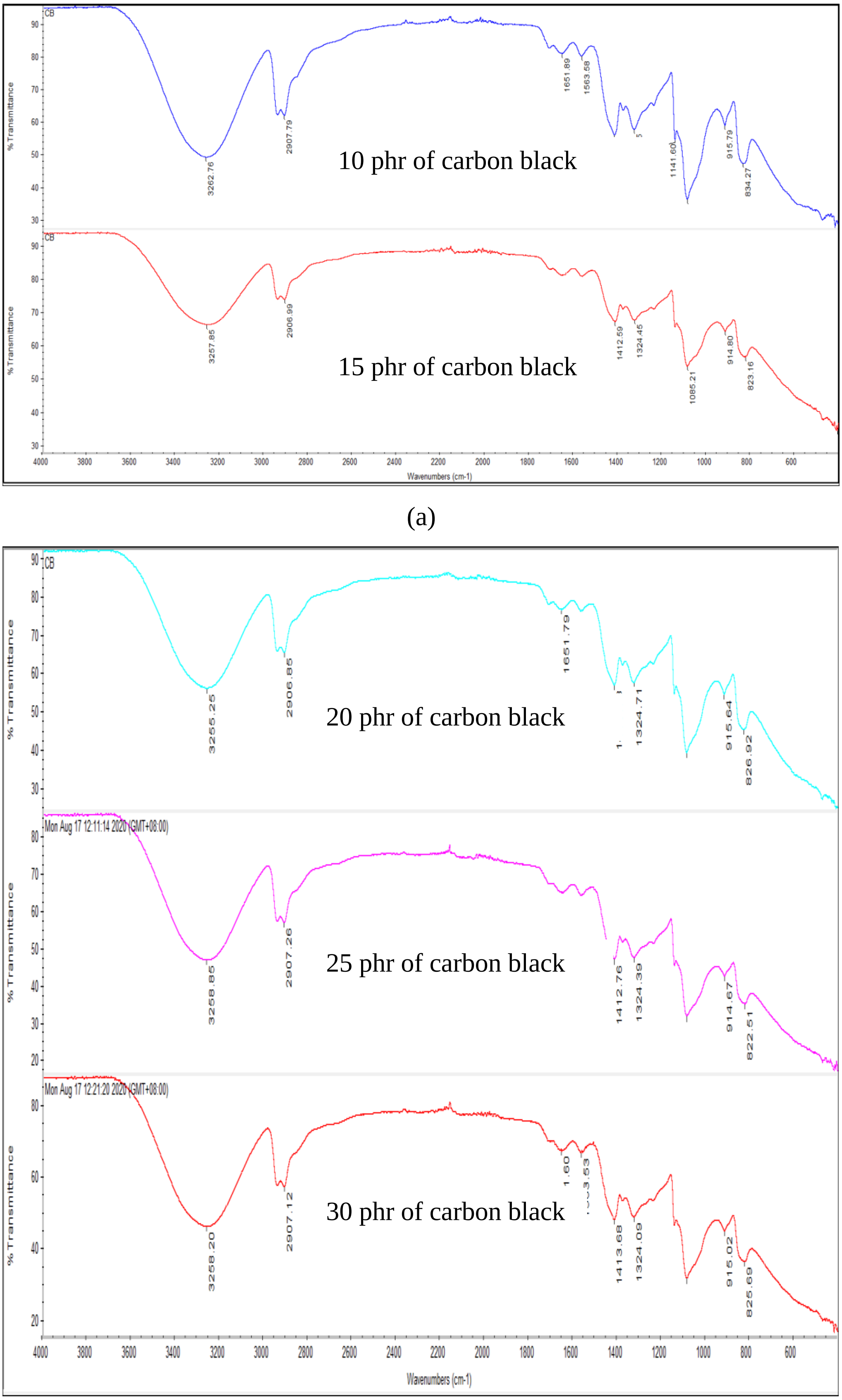
4.3.1 Fourier Transform Infrared (FTIR) Analysis for PVOH/Carbon Black composites

Figure 4.4 showed infrared spectrum of PVOH/CB composites with increasing amount of carbon black. By referring to Figure 4.4 to Table 4.1, there are two prominent peaks obtained in the FTIR spectrum of PVOH/CB composites at wave number bands of 3200 cm^{-1} to 3400 cm^{-1} and 2900 cm^{-1} to 3000 cm^{-1} respectively, which are the OH stretching and CH stretching. The changed of OH stretching and CH stretching represents that incorporation of carbon black into PVOH, which might alter the bonding strength within the lattice structure (Wen, et al., 2012). Furthermore, the effect of incorporating carbon black on the wave numbers of OH stretch and CH stretch of PVOH/CB composites were summarized in Table 4.1.

According to Table 4.1, the wavenumber of OH stretch of PVOH/CB composites has slightly decreased from 3262.76 cm^{-1} to 3258.20 cm^{-1} as the amount of the carbon black increased from 10 phr to 30 phr. However, for PVOH/CB composites with 25 phr of carbon black, the wave number of the OH stretch has increased from 3255.25 cm^{-1} to 3258.85 cm^{-1} when the carbon black increased from 20 phr to 25 phr. The increment in wave number of OH stretch indicates that the weakening effect of the hydrogen bonding (H-bond) inside the polymer matrix of PVOH/CB composites (Wang, et al., 2008). This also denoted that the addition of carbon black into PVOH has weakened the H-bond of polymer chains in PVOH. Furthermore, the good dispersion of HAP in PVOH has weakened the H-bond between the PVOH macro-molecular chains in polymer matrix by enhancing the interaction of carbon black and PVOH chains. In addition, the carbon black in PVOH could interact well with PVOH and induced interfacial adhesion effect with PVOH. Hence, this reducing of hydrogen bonding could further improve the strength of OH bond of PVOH chains (Bee, et al., 2018).

Besides that, it was observed that the wavenumber of C-H stretching remained almost constant at 2907 cm^{-1} as the amount of the carbon black increased from 10 phr to 30 phr. This indicates that the addition of carbon black does not have any effect to the CH stretch in the PVOH matrix.

Moreover, it could be observed in Table 4.1 that the wave number of CH stretch has slightly increased from 2906.85 cm^{-1} to 2907.26 cm^{-1} as carbon black increased from 20 phr to 25 phr, The can be due to the structure of PVOH matrix has been improved by strengthening the structure of PVOH. The excellent dispersion of carbon black in PVOH/CB composite could increase the specific surface area of carbon black interacted with the PVOH and hence induced the interfacial adhesion effect of carbon black and PVOH. This might further enhanced Van der Waals binding within PVOH and increased the changed in inter-molecular vibration, hence improved the CH stretch in PVOH (Bee, et al., 2018).



(a)

(b)

Figure 4.4: FTIR Spectrum of PVOH composites with different loading level of carbon black (a) 10 phr and 15 phr, (b) 20 phr , 25 phr and 30 phr

Table 4.1: Wavenumber of hydroxyl group (-OH) and C-H stretching of PVOH composites with different loading level of carbon black.

	Wavenumber, cm^{-1}	
Loading level of carbon black particles, phr	**O-H Stretching**	**C-H Stretching**
10 phr	3262.76	2907.79
15 phr	3257.85	2906.99
20 phr	3255.25	2906.85
25 phr	3258.85	2907.26
30 phr	3258.20	2907.12

4.4 Summary

In short, the addition of 10 phr carbon black particles in PVOH/CB composites had better results due to high tensile strength and Young's modulus. Besides, the surface morphologies of 10 phr carbon black added polymer bio-composites also showed a smooth and continuous surface when compared to others. The mechanical properties of PVOH/CB composites showed poorer results at higher amount of carbon black particles. This is because the poor dispersion of particles and the inducing effect of agglomeration, which can also observed from the surface morphologies results. Similarly, the wave number of OH stretch and CH stretch of PVOH/CB composite have slightly shifted to a lower wavenumber as amount of carbon black particles increased.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In this study, the effects of tyre derived carbon black particles added into PVOH have been intensively investigated. The tensile strength was decreased when adding 10 phr of carbon black in the PVOH blends. This is due to the agglomeration effect of carbon black which weaken the interaction and interfacial effect between carbon black and PVOH. Besides, Young's modulus was decreased continuously as the carbon black increased from 10 phr to 20 phr. PVOH composites with 10 phr of carbon black posed the highest tensile strength and Young's Modulus; while PVOH with 30 phr of carbon black showed the lowest tensile strength and Young's modulus. Furthermore, the surface morphologies of all PVOH/CB composites were conducted using SEM analaysis. The fractured surface of 10 phr carbon black added PVOH/CB composite was observed to pose less tearing effect at different phases. Besides, the cavities and the agglomerated particles was also found to be absence in the fractured surface of this sample. This observation had proved that the 10 phr carbon black added PVOH composite posed better result in mechanical properties. This observation is tally with the results of tensile strength and Young's modulus. Conversely, PVOH/CB composite with 30 phr of carbon black was observed to pose a brittle pattern, which had also the lowest tensile properties. The fractured surface of this samples was observed to be less continuity, high tearing effect at different phases and high occurrence of cavities and agglomerated particles. According to the FTIR analysis, the wave number of OH stretch was found to decrease the increasing amount of carbon black. This is due to the presence of carbon black which is hydrophobic behaviour could weaken the hydrophilic behaviour of PVOH and thus increased the hydrogen bonding between PVOH macromolecules. However, the wave number of CH stretch remains constant about 2907 cm^{-1} when increasing of carbon black loading level.

5.2 Recommendations for future work

It is recommended that other testing should be studied for better understanding of the properties of PVOH/CB composites. The thermal properties of the polymer composite are important for polymer nano-composite study. Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry DSC) are equipment can be applied for analysis of thermal properties of polymer nano-composites. TGA can used to determine the stages of thermal degradation, thermal stability and percentage of residue of polymer nano-composite. DSC can used to determine the melting temperature and enthalpy of melting of polymer nano-composites.

Besides that, a sample of PVOH/CB composite should also be studied for the XRD analysis and water absorption test. This is to make a comparison with the PVOH/CB composites in order to understand more details about the effect of adding carbon black particles.

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APPENDICES

APPENDIX A: Tensile Properties

Table A-1: Average results of Tensile Strength, Young’s Modulus for all PVOH/CB composites.

Sample	**Tensile Strength (MPa)**	**Young’s Modulus (MPa)**
P-110	99.24	27.84
P-115	76.81	23.20
P-120	74.17	18.06
P-125	86.63	24.45
P-130	67.11	19.20