PRODUCTION OF CONDUCTIVE POLYMER USING CARBON NANOTUBE

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this project and in my opinion, this project is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering.

Signature

Name of Supervisor: Dr Mohammad Dalour Hossen Beg

Position:

Date:

STUDENT'S DECLARATION

I hereby declare that the work in this project is my own except for quotations and summaries which have been duly acknowledged. The project has not been accepted for any degree and is not concurrently submitted for award of other degree.

Signature

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ABSTRACT

Polymeric based composite materials have large usage in the industrial scale, which involving improvement of mechanical properties to the alternation of thermal and electrical properties. Carbon nanotubes (CNTs) exhibit excellent mechanical, electrical, and magnetic properties as well as nanometer scale diameter and high aspect ratio, which make them an ideal reinforcing agent for high strength polymer composites. The objectives for this paper are to study the mechanical properties (tensile strength, young modulus and elongation) of conductive polymer, to study the effect of acid and heat treatment process towards the PLA mechanical and electrical properties, to identify effects of different weight percentage of untreated CNTs in PLA matrix and to compare the treated and untreated CNTs mechanical and electrical properties in PLA matrix. The samples prepared are categories into two types firstly using untreated CNTs with Polylactic acid (PLA) with different CNTs compositions and secondly sample prepared from two type of treated CNTs which were using thermal or heat surface treatment and acid surface treatment using hydrochloric acid and the untreated and treated CNTs are mixed into PLA polymer. Method used to produce sample for analysis was by passing through the material in an extrusion molding machine at specific temperature and screw speed followed by injection molding with specific pressure and temperature. The produce sample undergo several analysis and testing which were mechanical testing, Scanning Electron Microscope, Transgravimetric analysis and surface resistivity analysis. From the results it shows that untreated sample with 1.00wt % had higher tensile strength and elongation compared with acid and heat treated CNTs with 1.00wt% CNTs. Meanwhile, Transgravimetric analysis shows that the untreated 1.00wt% CNTs sample had the highest thermal degradation temperature compared to others sample. Surface resistivity analysis for comparing 1.00wt % treated and untreated sample it showed that acid treated sample has the lowest surface resistance with the value of 2.06826 Ohm/sq. The results obtained from the experiments is reliable and shows a pattern of data that can be used for further improving the research in obtaining the optimum amount of CNTs for better future research study. The data also shows that acid treatment with nitric acid is not proper for PLA polymer due to it degradation property when acid and basic impurities are present. In future improvement of research paper alternative treatment could be found in improving CNTs surface properties.

ABSTRAK

Bahan-bahan komposit berasaskan polimer mempunyai penggunaan yang besar dalam skala industri, yang melibatkan peningkatan sifat-sifat mekanik silih sifat haba dan elektrik. Nanotube karbon (CNTs) mempamerkan cemerlang sifat-sifat mekanikal, elektrik, dan magnet serta garis pusat skala nanometer dan nisbah aspek yang tinggi, yang menjadikan mereka ejen yang ideal memperkukuh bagi komposit polimer kekuatan tinggi. Objektif kertas ini adalah untuk mengkaji sifat-sifat mekanik (kekuatan tensil, modulus muda dan pemanjangan) polimer konduktif, untuk mengkaji kesan asid dan proses rawatan haba terhadap sifat-sifat mekanik dan elektrik PLA, untuk mengenal pasti kesan peratusan berat badan yang berbezaCNTs tidak dirawat di PLA matriks dan bandingkan CNTs dirawat dan tidak dirawat mekanikal dan sifat-sifat elektrik di PLA matriks. Sampel yang disediakan adalah kategori kepada dua jenis yang pertama menggunakan CNTs tidak dirawat dengan asid Polylactic (PLA) dengan komposisi CNTs berbeza dan kedua sampel yang disediakan daripada dua jenis CNTs dirawat yang menggunakan permukaan rawatan haba atau haba dan rawatan permukaan asid menggunakan asid hidroklorik dan tidak dirawat dan dirawat CNTs dicampurkan ke dalam PLA polimer. Kaedah yang digunakan untuk menghasilkan sampel untuk analisis adalah melalui bahan dalam mesin pengacuan penyemperitan pada suhu tertentu dan kelajuan skru yang diikuti oleh pengacuan suntikan dengan tekanan dan suhu tertentu. Sampel hasil menjalani beberapa analisis dan ujian yang ujian mekanikal, Pengimbasan Mikroskop Elektron, Transgravimetric analisis dan analisis kerintangan permukaan. Daripada keputusan, ia menunjukkan bahawa sampel yang tidak dirawat dengan% 1.00wt mempunyai kekuatan tegangan yang lebih tinggi dan pemanjangan berbanding dengan asid dan dirawat haba CNTs dengan CNTs% 1.00wt. Sementara itu, Transgravimetric analisis menunjukkan bahawa CNTs sampel% tidak dirawat 1.00wt mempunyai suhu degradasi haba tertinggi berbanding dengan sampel yang lain. Analisis permukaan kerintangan untuk membandingkan% 1.00wt dirawat dan sampel yang tidak dirawat, ia menunjukkan bahawa sampel asid yang dirawat mempunyai permukaan rintangan yang terendah dengan nilai 2,06826 Ohm / persegi. Keputusan yang diperolehi daripada uji kaji boleh dipercayai dan menunjukkan corak data yang boleh digunakan untuk meningkatkan lagi penyelidikan dalam mendapatkan jumlah optimum CNTs untuk kajian masa depan yang lebih baik. Data juga menunjukkan bahawa rawatan asid dengan asid nitrik tidak wajar untuk PLA polimer kerana harta degradasi IT apabila asid dan kekotoran asas hadir. Dalam peningkatan masa depan rawatan alternatif kertas penyelidikan boleh didapati dalam memperbaiki sifat-sifat permukaan CNTs.

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A	Fitted constant
c	The conductivity of the composites,
V	Carbon nanotube volume fraction,
Vc	Carbon nanotube volume fraction at the percolation threshold
˚C	Temperature measurement
ρ_s	Surface Resistivity
U	Voltage
L	Length
I_S	Surface Current
D	Diameter
M	Concentration
V	Volume

LIST OF ABBREVIATIONS

CNTs	Carbon Nanotube
PLA	Polylactic acid
SEM	Scanning Electron Microscope
UTM	Universal Testing Machine
TGA	Transgravimetric Analyzer

CHAPTER 1

INTRODUCTION

1.1.1 INTRODUCTION

Conducting polymer research dates back to the 1960s, when Pohl, Katon, and their coworkers, first synthesized and characterized semiconducting polymers. The discovery of the high conductivity of polysulfurnitride (SN)x, a polymeric material containing interesting electrical properties, was a step forward for research in conducting polymers. Historically, people have referred to polymer as plastic. Technically, plastic refers to the state in which a material is deformed plastically and does not return to its original underformed state (James C. Gerdeen et al., 2006). Polymeric based composite material has a large usage in the industrial scale, which involving improvement of mechanical properties to the alternation of thermal and electrical properties. It is also necessary to find and alternatives to produce conductive polymer using carbon nanotube due to market demand and recent abundant research done on carbon nanotube as nano-filler.

Conductive composite is well fitted to carbon nanotube (CNTs) this is due to several factors such as exhibit high aspect ratio and high conductivity. Carbon nanotubes (CNTs) exhibit excellent mechanical, electrical, and magnetic properties as well as nanometer scale diameter and high aspect ratio, which make them an ideal reinforcing agent for high strength polymer composites. (Nanda Gopal Sahooa et al, 2010)

Percolation theory predicts that there is a critical concentration at which composites containing conducting fillers in insulating polymers become electrically conductive.

According to percolation theory Eq (1.1):-

$$c = A(V-V_c) \tag{1.1}$$

c = the conductivity of the composites,
V = CNT volume fraction,
Vc = CNT volume fraction at the percolation threshold
(Percolation threshold has been reported to ranging from 0.0025 wt% to several wt % - Sandler JKW et al)

A= fitted constant.

The percolation threshold for the electrical conductivity in polymer-CNT nanocomposites depends on dispersion, alignment, aspect ratio, degree of surface modification of CNTs, polymer types and composite processing methods.

This research has chosen polylactic acid (PLA) compared with other biodegradable polymer such as poly(ε-caprolactone) (PCL) due the several factor. PLA compared to other biodegradable polymer are higher biodegradability and better mechanical properties such as tensile strength, results in a material whose properties make it easier to process and diversifies its possible applications such as chemotherapeutic (E. Laredo et al, 2010).

1.2 PROBLEM STATEMENT

Recent, industrial grow had demanded the research on conductive polymer with higher conductivity and improve properties of polymer polymeric films are inherently insulating, they can become charged during ground-based manufacturing and handling as well as in space due to the orbital environment. The material can then behave like a capacitor and discharge in a single event causing considerable damage to surrounding materials and electronics on the vehicle.

The current state-of-the-art to impart electrical conductivity while maintaining a low and high optical transparency has been through the use of conductive coatings such as indium–tin oxide (ITO). While exhibiting high surface conductivity, these coatings are rather brittle and make handling difficult. Once the coating is broken (cracked) by handling or on orbit, the conductive pathway is lost. Conductive Polymer is the best solution for this problem due high transparency.

1.2 RESEARCH OBJECTIVE

In general, this research is related towards finding properties of mechanical properties conductive polymer (PLA/Carbon Nanotube) and it can be distinguished into four particular scopes:

i. To study the mechanical properties (tensile strength, modulus of elasticity and yield) of conductive polymer.
ii. To study the effect of acid and heat treatment process towards the PLA mechanical and electrical properties.
iii. To identify effects of different weight percentage of untreated CNTs in PLA matrix.
iv. To compare the treated and untreated CNTs mechanical and electrical properties in PLA matrix

1.3 SCOPE OF STUDY

In achieving, the first objective which is to study the mechanical properties of conductive polymer several test will be tested towards the specimen such as simple stress and strain behavior (William D. Callister et. al., 2008). From this test the stress strain behaviors can be plotted in graphical view where the effect of stress and strain can be clearly seen.

Meanwhile, in achieving the second objective the treatment process related in treating the CNTs using acid and heat had been identified. By using this treatment method the sample contain CNTs which are treated are prepared.

Third objective can be achieved by making different weight percentage of CNTs which are 0.5wt%, 1.0wt% and 1.5wt% in PLA matrix. The different composition of CNTs in PLA matrix will be studied by comparing its mechanical properties and electrical properties by using results from independent tested results.

Final objective is achieved, by comparing the same weight percentage of CNTs in the acid, heat and untreated sample to obtain the needed data. The data obtain from this three type of sample will give a better comparison for sample which has undergo CNTs treatment process and untreated CNTs.

1.4 RATIONALE & SIGNIFICANCE

Rationale of this research is that it can improve the capabilities of biodegradable polymer nowadays not only to function as plastic bag and everyday usage material. The properties of PLA which are environmental friendly give an upper hand in selecting a proper polymer matrix in the production of conductive polymer.

This research will identify the effects of CNTs on the polymer matrix. Furthermore, this research will be able to identify the effect of treated CNTs towards PLA matrix which will give an overview on the future improvement of conductive polymer application and usage.

Improving PLA properties also is another significant outcome that can be expected from this research. This is due to the improvement that can be seen from the usage of CNTs in PLA matrix will give and improvement not only on application of PLA nowadays but also in future.

CHAPTER 2

LITERATURE REVIEW

2.1 POLYMER

Polymer can be defined as material which contains many unit of monomer example polyethylene which builds from many unit of ethylene (monomer) (James C. Gerdeen et al., 2006). The most known properties are function as an insulator and has high melting and boiling point. (Mohd Hamzah Harun et al, 2007). From description, of this polymer that conductive polymer is a venture of chemical engineering as said in Mohd Hamzah et al.(2007) article about scientists discovery on electrical properties of conjugated polymer that called ‘polyacetylene’ which become highly electrically conductive after undergoing a structural modification process called doping structural modification process called doping.

Several properties of poly(lactic-acid), has made it one of the most attractive and useful biodegradable polymer, such as biodegradable, biocompatible and compostable polyester derived from renewable resources such as corn, potato, cane molasses and beet sugar. It is one of the most promising environmentally friendly thermoplastics. (K. Chrissafis, 2010).

Polylactide, obtained from the polymerization of lactic acid (2-hydroxypropionic acid, CH3–CH(OH)–COOH), is also known as polylactic acid, PLA. It has recently entered worldwide in the market for use as a biodegradable plastic .Due to the comparable mechanical properties with standard thermoplastics such as polyethylene terephthalate (PET) and polyvinyl chloride (PVC), PLA could represent a good candidate for the replacement of petrochemical thermoplastics. (Saveria Santangelo et al., 2010).

PLA also a thermoplastic polymer, aliphatic polymer and can be derived from renewable sources such as starch and is a sustainable alternative to petrochemical-derived products. PLA has been found to have good stiffness and strength and is being used in several applications, such as food packaging, water and milk bottles, degradable plastic bags as well as in automotive applications. The products made from PLA are bio-degradable and are found to fully disappear in less than 30 days in ideal conditions (Mehdi Jonoobi et al.,2010).

2.2 CARBON NANOTUBE

2.2.1 Introduction Carbon Nanotube

Carbon Nanotube used as nano-fillers in existing polymeric materials to both dramatically improve mechanical properties and create highly anisotropic nanocomposites. They can also be used to create electrically conductive polymers and tissue engineering constructs with the capacity to provide controlled electrical stimulation.

Carbon nanotubes (CNT) are unique, one-dimensional macromolecules, whose outstanding properties have sparked an abundance of research since their discovery in 1991 (S.K. Smart et al., 2005). Single-walled carbon nanotubes (SWCNT) are constructed of a single sheet of graphite (diameter 0.4– 2 nm), while multi-walled carbon nanotubes (MWCNT) consist of multiple concentric graphite cylinders of increasing diameter (2–100 nm) (S.K. Smart et al., 2005). Both SWCNT and MWCNT have high tensile strength, ultra weight and have excellent thermal and chemical stability. Besides that, they also possess semi-conductor electronic properties and metallic properties (S.K. Smart et al., 2005).

2.2.2 Biocompatibility of carbon nanotubes

In these studies, it shows that CNT biocompatibility is shown from the the unique electronic properties of CNT for used in neural applications focusing particularly on neurite extension (S.K. Smart et al., 2005). Neurite extension biocompatibility shows that positively charged ethylenediamine-MWCNT producing the most neurite extension due to the loosely ionic charge (S.K. Smart et al., 2005).

Biocompatibility also shown, in utility of CNT-containing materials as bone biomaterials, by examining the adhesion and function of bone-forming osteoblast cells osteoblast proliferation on nanocomposites of CNT under alternating current stimulation was also investigated. S.K Smart et al.(2005) had identified a research that fabricated conductive polylactic acid (PLA)/MWCNT nanocomposites at 10, 15 and 20% (synthesized via electric arc-discharge) whereby the osteoblast cells were seeded onto the surface and then exposed to alternating current stimulation. Control samples (PLA/MWCNT nanocomposite films) were run without electrical stimulation. Their results showed an increase in osteoblast proliferation and extra-cellular calcium deposition on the nanocomposite.

2.3 METHOD FOR POLYMER PRODUCTION

2.3.1 In situ polymerization processing

Over the last 5 years in situ polymerization in the presence of carbon nanotubes has been intensively explored for the preparation of polymer grafted nanotubes and processing of corresponding polymer composite materials. The main advantage of this method is that it enables grafting of polymer macromolecules onto the walls of carbon nanotubes. In addition, it is a very convenient processing technique, which allows the preparation of composites with high nanotube loading and very good miscibility with almost any polymer type. This technique is particularly important for the preparation of insoluble and thermally unstable polymers, which cannot be processed by solution or melt processing. (Jonathan et al., 2006). In situ polymerization also has the capabilities in improving the mechanical properties of polymer.

Kumar et al.,2010 have synthesized new ultra-strong poly (pphenylene benzobisoxazole) (PBO) composites in the presence of single-wall carbon nanotubes (SWNTs) in poly(phosphoric acid) (PPA) by in situ PBO polymerization. In general, in situ polymerization can be applied for the preparation of almost any polymer composites containing carbon nanotubes which can be non-covalently or covalently bound to polymer matrix. Non-covalent binding between polymer and nanotube involves physical adsorption and wrapping of polymer molecules through van der Waals and p–p interactions.

2.3.2 Covalent Functionalisation and Polymer Grafting Of Nanotubes

Single-Walled carbon nanotube (SWCNs) covalent functionalisation and surface chemistry is an essential factor for nanotube processing and application. (Jonathan et al). Carbon nanotube and the polymer matrix usually has small amount bonding attraction this is due to the relative smooth graphene surface of nanotube. Nanotube, in particular SWNTs, is typically held together as bundle which resulting in poor dispersion in polymer matrices. (Jonathan et al,.2006). Covalent functionalisation is expected to resolve this problem by either modification of surface-bound carboxylic acid groups on the carbon nanotube or direct addition of reagent towards the wall of carbon nanotubes. (Jonathan et al., 2006).

Furthermore, this method has two strategies which are “grafting from” and “grafting to”. The ‘‘grafting from’’ method is based on the initial immobilization of initiators onto the nanotube surface followed by the in situ polymerization of appropriate monomers with the formation of the polymer molecules bound to the nanotube. The advantage of this technique is that polymer–nanotube composites with quite high grafting density can be prepared. However, this method requires a strict control of amounts of initiator and substrate as well as a control of conditions for polymerization reaction. (Jonathan et al., 2006).

The ‘‘grafting to’’ approach is based on attachment of already desired end-functionalised polymer molecules to functional groups on the nanotube surface via different chemical reactions. An advantage of this method is that preformed commercial polymers of controlled mass and distribution can be used. The main limitation of the ‘‘grafting to’’ technique is that initial binding of polymer chains sterically prevents diffusion of additional macromolecules to the surface leading to low grafting density. Also only polymers containing reactive functional groups can be used. (Jonathan et al., 2006).

“Grafting from” strategies has been used in modification of SWNTs with polyethylene (PE) by in situ Ziegler–Natta polymerization. (Tong et al.,2004). In this, modification the SWNTs was functionalized with the catalyst (MgCl2/TiCl4), and then the ethylene was polymerized thereby giving PE grafted SWNTs, which were mixed with commercial PE by melt blending. (Tong et al., 2004). Meanwhile, “grafting to” strategies was used in polymer containing reactive functional groups. For example, Bhattacharyya et al. have developed new fully integrated nanotube–PVA composite materials through the functionalisation of MWNTs by covalently attaching ferritin protein molecules onto the surface of nanotubes.

2.4 APPLICATIONS

2.4.1 Designing sensitive electrochemical sensors

This is whereby conductive polymer, such as Polyaniline(PANI) used to design sensitive electrochemical sensors. It was observed that with an increase of the nanotube concentration, the conductivity of PANI/nanotube films and the current level in the metal-semiconductor devices increase, even at an elevating temperature condition. (Kin-tak Lau et al., 2006)

2.4.2 Power application

Power application, uses conductive polymer to ensure safety and efficient in their system such as proton exchange membrane (PEM) fuel cell, polymeric solar cells, polymeric solar cells LiC batteries, and thermionic emitters. (Kin-tak Lau et al., 2006)

2.4.3 Biomedical material and devices

Conducting polymer can change their property by incorporation of ions and solvent which is a most dependent device to measure conductivity, it is possible to develop and market ion-specific sensors based upon conducting polymers (Mohd Hamzah Harun et al., 2005). Biomedical is a field whereby carbon nanotube hugely used in major area such in the production of biomedical material and devices such as biosensors, drug and vaccine delivery devices. (S.K. Smart et al., 2005)**.**

Conducting polymers also the capabilities in changing electrical energy to mechanical energy example is It is therefore possible for conducting polymers to convert electrical energy into mechanical work oxidation-induced strain of polyaniline and polypyrrole-based actuators has been reported, and the first ‘self-contained’ actuators (Mohd Hamzah Harun et al., 2005).

2.4.4 Radio Direction and Ranging (RADAR) Application

RADAR uses electromagnetic waves that bounce off a particular target and are collected by a receiver, which analyzes the signal and determines the range, direction, and speed of the object in question (Mohd Hamzah Harun et al.,2007). Reflections occur whenever there is a sharp impedance difference between the medium (usually air) and the object. Metals tend to re-radiate (reflect) the incoming signal. Conducting-polymer camouflage works a little differently, in that it reflects back in a way that it has more continuously variable impedance. A conducting polymer textile used for camouflage has no sharp edges or wings and tends to appear indistinguishable from the surrounding hills and trees and absorbs more than 50% of incident microwave radiation. (Mohd Hamzah Harun et al., 2007). Conducting polymers as radar absorbers in antennas, camouflage, and other types of shielding are of interest to the military.

2.5 LATEST FOUNDING ON CONDUCTIVE POLYMER

Latest founding about conductive polymer are told in E. Laredo et al.(2010) which had spoken about a polymer blend which conduct Direct Current (DC) and Alternating current (AC) electrical conductivity of bionanocomposites based on the immiscible polymer blend poly-(ε-caprolactone)/polylactide (PCL/PLA, w/w 70/30), loaded with multiwall carbon nanotubes (CNT) . From this research, selective location of the CNT in the PCL phase is responsible for the significant improvement of the mechanical properties of this biodegradable blend and its electrical properties are also quite promising for future applications (E. Laredo et al., 2010).

Another founding which is related to conductive polymer is from Saveria Santangelo et al.(2011), in this article they have written on exploring the preparation of novel biodegradable PLA nanocomposites by the use of carbon nanotubes (CNT) and clay as filler, and of evaluating how their mass and electrical transport properties change with respect to the unfilled polymer. From this, article they had concluded that results obtained suggest that clay and CNT exert a synergistic action in improving the polymer properties such as electrical conductivity (Saveria Santangelo et al., 2011.).

2.6 COMPARING PHYSICAL PROPERTIES OF METALS, INSULATORS AND CONDUCTING POLYMERS.

All materials can be divided into three main groups: conductors or metals, insulators and semiconductors. They are differentiated by their ability to conduct or the ability to allow the flow of current. Generally, conducting polymers are classified as semiconductors although some highly conducting polymers, such as polyacetylene, fall into metal range. Table 1 compares some of the physical properties of insulators, semiconductors and conductors. (Mohd Hamzah Harun et al., 2007)

Table 2.1: Comparison of physical properties of metals, insulators and conducting polymers.

Property	**Conducting polymers**	**Metals**	**Insulators**
Electrical Conductivity (S/cm)	$10^{-11} - 10^{3}$	$10^{-4} - 10^{6}$	$10^{-20} - 10^{12}$

Carriers	Electrons of conjugated double bonds	Valence electrons of half filled band	–
Concentration of carriers per cm3	$10^{-12} - 10^{19}$	$10^{-22} - 10^{23}$	–
Effect of impurity	Impurities of 0.1 – 1% change conductivity by 2 to 3 orders of magnitude	Effect comparatively slight	Strong effect
Magnetic properties	Paramagnets	Ferro and diamagnets	Diamagnets

Source: Mohd Hamzah Harun et al.(2007)

2.7 CARBON NANOTUBE TREATMENTS PROCESS

2.7.1 Introduction

Novelty of Multi-walled (MWCNT) carbon nanotube in their mechanical, electrical and gas storage properties has made them an attraction towards industry application. However, the properties in MWCNT are restricted by their surface properties which are hydrophobic and inert in nature so MWCNT is unfavorable for fabrications and chemical reaction which restrict their applications. Functionalizing the MWCNT surface is an attractive method for improving MWCNT dispensability in the solution wet ability for polar solvents, and compatibility with polymers in composites. (Yu-Chun Chiang et al, 2011)

Van der Waals attraction between CNTs usually forms a cluster which causes it difficult to be dispersed and align in polymer matrix. An effective way in ensuring the CNTs has better dispersion and aligment in polymer matrix is by the use of functionalization of CNTs which will stabilize the CNTs. (Nanda Gopal Sahoo et al., 2010)

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

In this, method use in achieving the purpose of this research will be by using extrusion molding and injection molding. Meanwhile, analysis the sample will be done in four types of analysis which are tensile test, thermogravimetric Analysis (TGA), surface resistivity measurement and scanning electron microscope (SEM) analysis. Raw material will be used in this research are polylactide acid (PLA), and carbon nanotube (CNT) The research will be done in a different composition of untreated CNTs in the PLA matrix which are 0.5% wt, 1.0 %wt and 1.5wt% and treated CNTs in 1.00wt % . Sample will be build based from the composition of CNT in PLA matrix and all the sample will undergo analysis to identify its properties of each independent sample. Figure 3.1 shows the overall workflow that has been done in obtaining results.

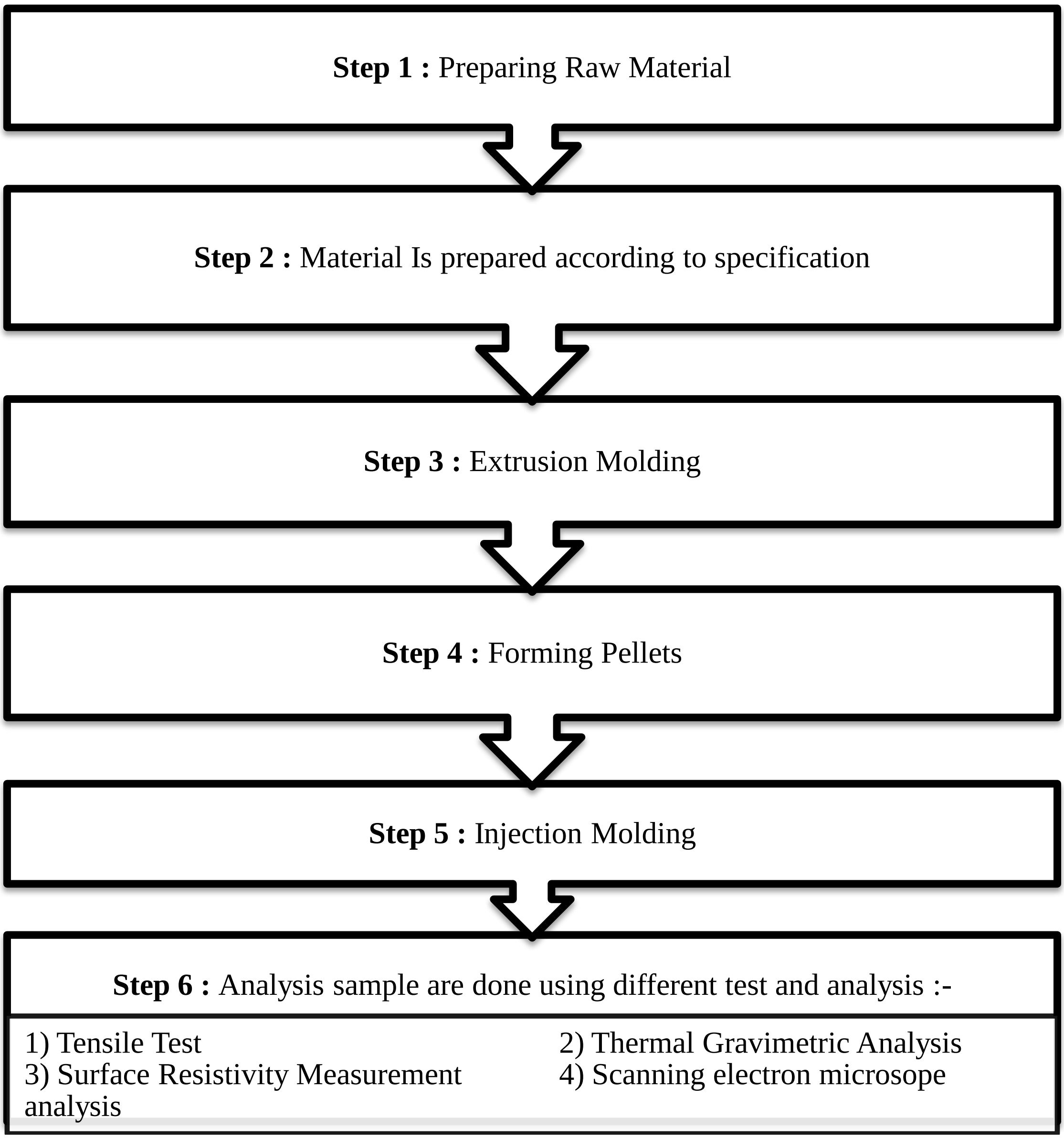


Figure 3.1: Overall Research Methodology Flow work

3.2 MATERIAL

3.2.1 Carbon Nanotubes (CNTs)

Carbon nanotubes are the main material for this research. The Carbon Nanotubes purchased from Sigma Alderich. Type of CNTs used was Multiwall Carbon Nanotubes, Which has high purity (>90%). This carbon nanotubes (CNTs) will be treated in acid solution, which is found in the journal. The CNTs is not dispersed in the solution because highly stable. Furthermore based from Nanda Gopal Sahooa et al.

(2010) explains that CNTs is an ideal reinforcing agent due to their properties which has excellent mechanical, electrical and magnetic properties.

3.2.2 PLA

G.Z. Papageorgioua et al. (2010) explain that PLA is high molecular weight polymer which is produce via the lactide ring-opening polymerization and has excellent mechanical properties and bio-safety. Furthermore, the PLA is biodegradable which would be an environmentally friendly polymer. PLA is the matrix than the CNTs dispersed.

3.2.3 Nitric Acid, HNO_3

According to Yu-Chun Chiang et al. (2011) treatment of CNTs with nitric acid will functionalize the MWCNTs. So Nitric acid is used to improve surface of CNTs. The Nitric acid is available in Store 3 in Laboratory of Chemical Engineering and Natural Resources, UMP. This Nitric acid is in high concentration, 16.4M (69%). Nitric acid is highly corrosive, so it needs to conduct with highly protection, which is wearing gloves. Below are the physical properties of HNO_3:

Table 3.1: Properties of Acid Nitric

Molar mass	63.01 g mol^{-1}
Appearance	Colorless liquid
Density	1.5129 g cm^{-3}
Melting point	-42 ^{o}C
Boiling point	83 ^{o}C (68% solution boiling at 121 ^{o}C)
Solubility in water	Completely immiscible

3.3 EQUIPMENTS

3.3.1 Extrusion Molding

Extrusion molding is will be the first step in production of conductive polymer using PLA and CNT. This extrusion is a process whereby the temperature of the inserted mixture will be increased gradually as the mixture gone thru the barrel. In this step of experiment the raw material will be inserted into the extruder feed hopper and the temperature of heater will be set in a range of 150 ˚C to 180 ˚C and the extruder screw speed is set in a range of 75 rpm to 120 rpm. Then finally, sample is obtained when mixed raw material entered the extrudate and form tube shape. The sample is than rested at certain temperature before undergoing pelletizing. This sample will undergo pelletizing whereby the tube sample is from into a pellet size to be inserted into injection molding process.

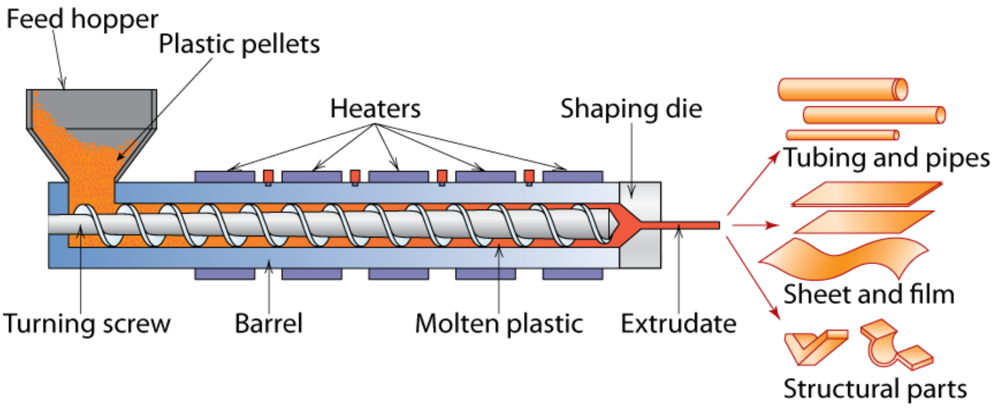


Figure 3.2: Extrusion Molding (Model)

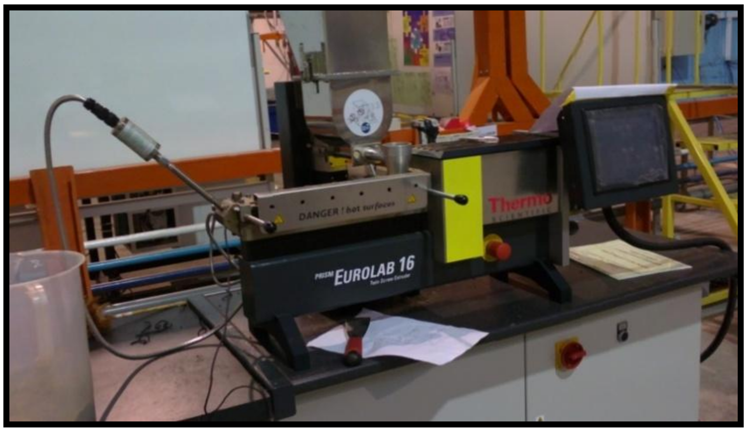
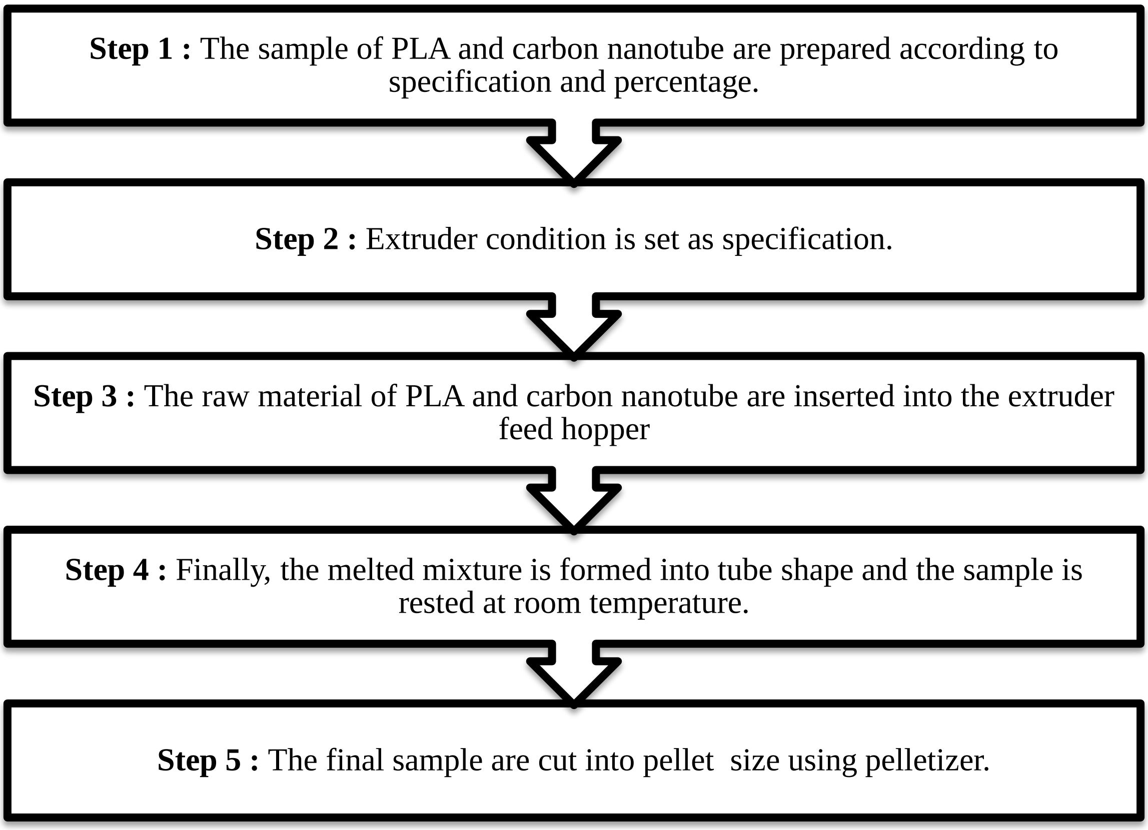


Figure 3.3: Extrusion Molding (Universiti Malaysia Pahang)



Step 1 : The sample of PLA and carbon nanotube are prepared according to specification and percentage.

Step 2 : Extruder condition is set as specification.

Step 3 : The raw material of PLA and carbon nanotube are inserted into the extruder feed hopper

Step 4 : Finally, the melted mixture is formed into tube shape and the sample is rested at room temperature.

Step 5 : The final sample are cut into pellet size using pelletizer.

Figure 3.4: Flow Work of Extrusion Molding

Figure 3.5 : Example of Pellet sample obtain after extrusion process

Figure 3.6 : Example of Pellet sample obtain after extrusion process (treated sample)

3.3.2 Injection Molding

Injection molding is second step in production of conductive polymer using PLA and CNT. Injection molding will give a better dispersion of carbon nanotube in PLA matrix. In this step of experiment the raw material will be inserted into the injection molding feed hopper and the temperature of heating chamber in a range of 160˚C to 190 and hydraulic pressure is set in range of 90 bar to 100 bar. Final step, is to inject the melted sample into molding cavity whereby the final sample are obtain before sample analysis undergo. The sample will be rested in room temperature before undergo analysis.

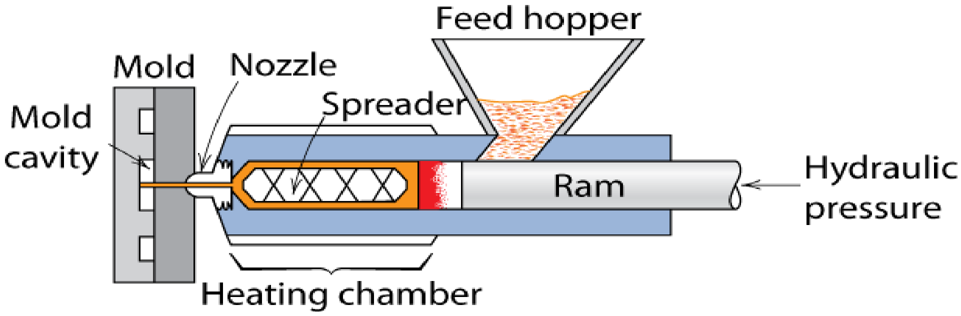
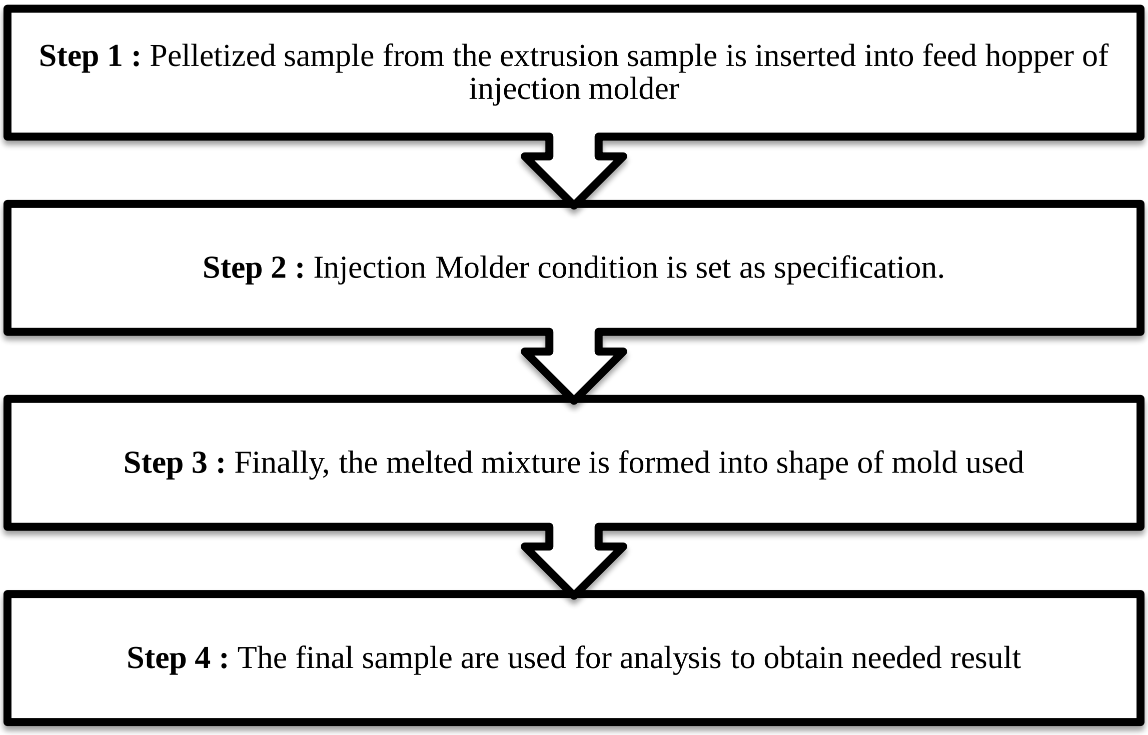


Figure 3.7: Injection Molding (Model)

Figure 3.8: Injection Molding Machine (University Malaysia Pahang, Pekan Campus, Mechanical Engineering Faculty)



Step 1 : Pelletized sample from the extrusion sample is inserted into feed hopper of injection molder

Step 2 : Injection Molder condition is set as specification.

Step 3 : Finally, the melted mixture is formed into shape of mold used

Step 4 : The final sample are used for analysis to obtain needed result

Figure 3.9: Flow Work of Injection Molding

Figure 3.10: Example of PLA/CNT Composite Sample from Injection Molding process

Figure 3.11: Example of Pure Polylactic –acid (PLA) Sample after Injection Molding

3.3.3 Universal Testing Machine (UTM)

UTM is used to obtain the data related to mechanical property of sample such as tensile strength, Young Modulus and Elongation of sample. There are several basic components in UTM machine which are:-

(i) Load Frame – the strong supporter for the machine.

(ii) Load Cell – A load transducer, which mean place where the load is measured.

(iii) Cross Head - A movable crosshead that can move up and down in constant speed. It can be change manually, by pressing the up and down button on the controller.

(iv) Output Device – UTM in Chemical Engineering lab use computer as output device. Place where the result was recorded.

(v)

The procedure for obtaining analytical data using UTM is by identifying the American Standard Testing Measurement (ASTM) which will be used to create method in UTM and execute the analysis and obtaining result.

Figure 3.12: Universal Testing Machine

3.3.4 Scanning Electron Microscope

Scanning Electron Microscope (SEM) use to find or to observe the dispersion of CNTs in different sample that has been prepared. SEM also shows the fracture surface after the mechanical testing using UTM is done.

Figure 3.13: Scanning Electron Microscope (SEM)

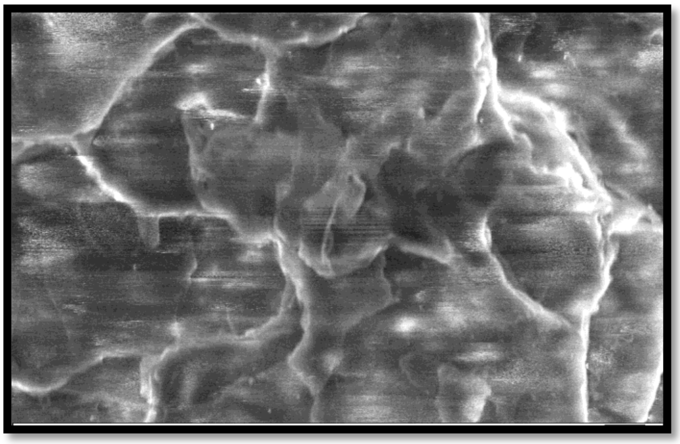


Figure 3.14: Example of Surface Image obtain from SEM

3.3.5 Thermogravimetric Analyzer

Function of thermogravimetric analyzer in this research is to determine the thermal decomposition temperature. From this data the sample can be identified it capabilities and defects in each sample which can be seen from the graph.

Figure 3.15: Thermogravimetric Analyzer

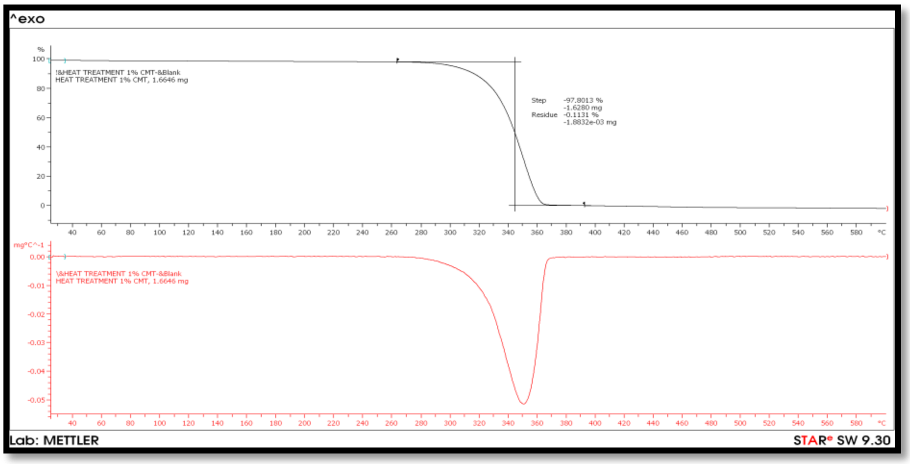


Figure 3.16: Example of Result obtained from TGA analysis

3.3.6 Surface Resistivity Measurement

William A. Maryniak et al. (2003) has used a concentric Ring Probe Technique in measuring Surface Resistivity and Surface Resistance. The surface resisitivitiy is measured by the ratio of DC voltage,U drop per unit length,L to the surface currentI_s per unit width,D. The formula is as expressed in equation Eq (3.1) :-

$$\rho_s = \frac{\frac{U}{L}}{\frac{I_S}{D}} \qquad (3.1)$$

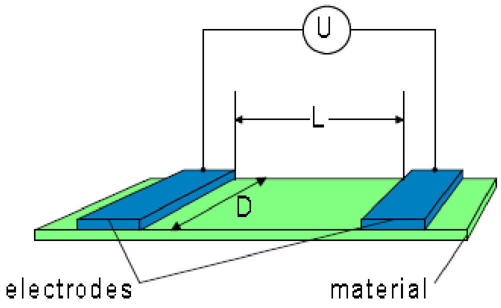


Figure 3.17: Basic setup for surface resistance and surface resistivity measurement

This setup used to find the surface resistivity (Ohm/square) for the entire sample. The setup for the apparatus is done according to Figure 3.15 and then the surface current is measured to identify the surface resistivity of sample.

3.4 SAMPLE PREPARATION

Type of sample used has been divided into two types which are treated carbon nanotube sample and untreated carbon nanotube sample. The untreated carbon nanotube sample has been divided into 3 types in different composition which are 0.5 wt%, 1.0wt% and 1.5%. Meanwhile the treated CNTs sample had been divided into two type of treatment process which are heat treatment and acid treatment of carbon nanotube. The treated sample has the same weight percentage of CNTs which are 1.00wt %.

3.4.1 Preparing Untreated CNTs Sample

The untreated sample is prepared using CNTs which has not undergone any kind of treatment process and are mixed to PLA sample. The amount of CNTs and PLA is determined using weight basis or weight percentage.

Preparing Sample containing 0.50wt%, 1.0wt% and 1.5wt% according to procedure below:-

i) Weight the amount of CNTs needed according to the weight percentage.

ii) Weight the amount PLA required according to weight percentage.

iii) Mixing are done towards the two raw material in a beaker and ensuring that the CNTs and PLA phase a homogeneous or well mixed.

iv) Well mixed sample can be used for the extrusion process in the production of pellets.

3.4.2 Preparing Treated CNTs Samples

3.4.2.1 Acid Treatment Procedure for CNTs

Carbon nanotubes is highly stable and cannot dispersed in the organic or inorganic solution. In order to counter this properties of CNTs, it will undergoes for an acid treatment. Not only acid treatment can be done to help CNTs dispersed in the Urea Formaldehyde, but other methods had been suggested which is using surfactant. The best method and easy to find the equiptment is acid treatment usnig acid nitric. Below is the procedure that had been followed:

a) Prepare the apparatus needed which are:
    (i) Beaker of 1L
    (ii) Measuring cylinder
    (iii) Hot plate and magnetic stirrer
    (iv) Magnet
    (v) pH meter
    (vi) 2M of Acid Nitric, HNO_3
    (vii) Aluminium foil

b) Preparing 1000 mL of 2M of Acid Nitrics.

    (i) The Acid Nitrics inside Store 3 is in high concentration, which is 16.4 M. We need to used only for 2 M of HNO_3**.** So**,** we need to dilute the high concentration of HNO_3 by using this equation expressed as below Eq. (3.2)**:**

$$M_1V_1 = M_2V_2 \tag{3.2}$$

(ii) Calculation had been made and can be refferd in Appendix B1. The volume of acid needed and water needed to dilute into 1000 mL of 2M of Nitric acid are:

$$V_{HNO_3} = 122mL\ of\ acid$$
$$V_{water} = 878mL\ of\ water$$

c) Prepare 10 g of CNTs and deep it in the 2M of HNO_3. This treatment must be followed by continuous stirring and heating around 4 hours. This steps must be done inside fume hood and the acid solution must be covered with aluminium foild.

Figure 3.18: Stirring and heating the CNTs

d) After 4 hours treatment, let the acid solution cooled down until room temperature.

e) Then check the pH of the acid solution and dilute it with tap water until reach pH 7.

f) Filtrate the CNTs using vacuum pump. The filtrate paper used is around 125mm diameter of pores.

Figure 3.19: Vacuum filter

3.4.2.2 Heat Treatment Procedure for CNTs

This treatment process is done by heating the CNTs sample in a furnace up to 500℃. This heating temperature range is obtained based form the research paper from Alejandro De Falco et al. (2009). Alejandro De Falco et al. (2009) also explain the usage of thermal treated MWCNTs to be added to a polymer which is polystyerene. By using this thermal treatment the heat treatment for CNTs had been done.

Figure 3.20: Glass Furnace at Universiti Malaysia Pahang

CHAPTER 4

RESULT AND DISCUSSION

4.1 INTRODUCTION

4.2 MECHANICAL PROPERTIES OF PLA/CNT COMPOSITE

4.2.1 Tensile Strength Analysis

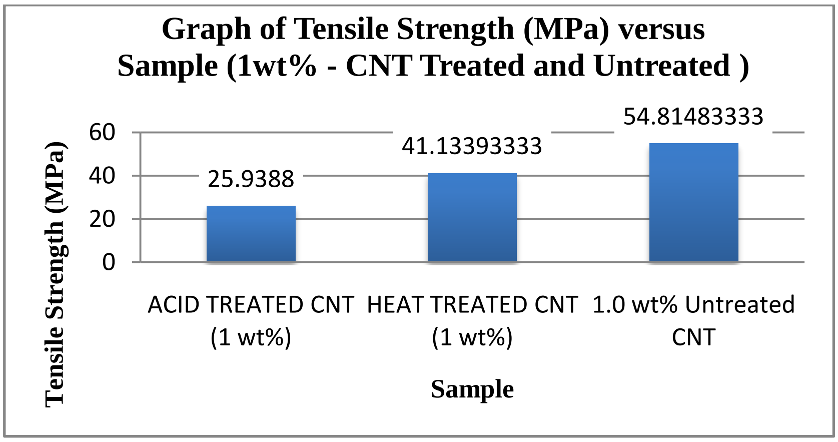


Figure 4.1: Graph of Tensile Strength (MPa) versus Sample (1wt% - CNT Treated and Untreated)

From the figure above it shows that the tensile strength (MPa) of untreated sample is much higher compared to the treated sample. The data also shows that the percentage different in tensile strength between untreated and heat treated is 24.96% and 52.68% the percentage different comparing acid treated and untreated sample. The composite of CNTs in all three samples are in the same weight percentage which is 1.00wt%. So the treatment process which has been executed shows that the tensile strength of sample reduces drastically which could be caused by the inappropriate treatment for PLA. Furthermore, a study done by Defeng Wu et al.(2008) suggested that the acid treated CNTs could affect the PLA matrix if acidic or basic impurities is presents. The reduce in the tensile strength of treated sample also due to it has better dispersion and interaction with the PLA matrix compared to treated carbon nanotube sample.

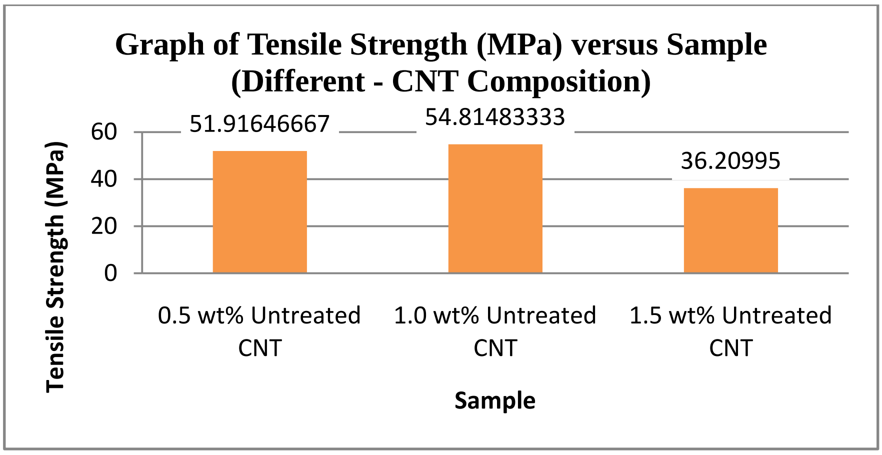


Figure 4.2: Graph of Tensile Strength (MPa) versus Sample (Different - CNT Composition)

This graph shows the tensile strength of sample with differebt amount of untreated carbon nanotube which are 0.5wt% , 1.0 wt% and 1.5wt%. From the figure it also shows that the 1.0 wt% CNTs in PLA matrix shows the highest tensile strength, secondly 0.5wt% and the weakest tensile strength is obtain from 1.5wt%. The percentage of different in tensile strength for comparing 1.0wt %CNTs with 0.5wt% is around 5.29wt% and for 1.0wt % CNTs with 1.5 wt% is 33.94% different. This shows that the amount of CNTs in a PLA matrix is affecting the amount of tensile strength than can be obtain for a sample.

4.2.2 Young Modulus (MPa) Analysis.

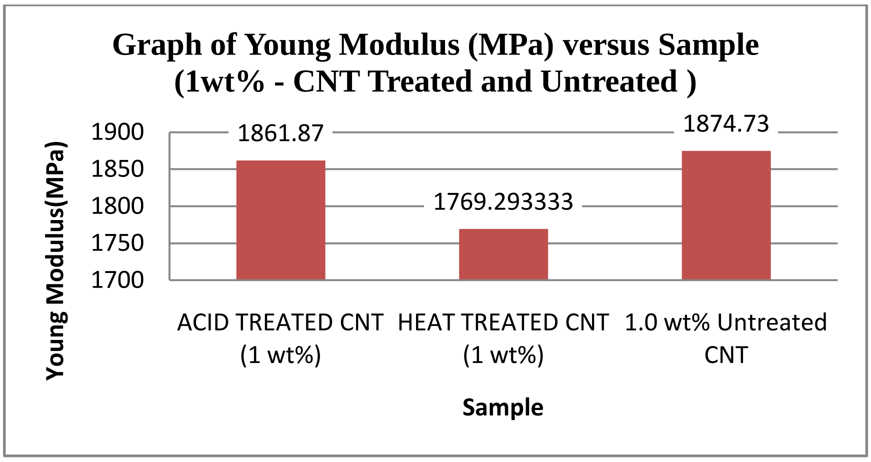


Figure 4.3: Graph of Young Modulus (MPa) versus Sample (1wt% - CNT Treated and Untreated)

Figure is showing the Young Modulus that obtained from the sample containing same amount of CNTs which are 1.0 wt% in untreated and treated sample of CNTs. The highest Young Modulus shown by the untreated sample which is 1874.73 MPa followed by acid treated sample and the lowest Young Modulus obtained by the heat treated sample. The comparison of higher young modulus for acid treated compared to heat treated this is due to the factor that better dispersion of acid treated CNTs in PLA matrix compared to heat treated CNTs.

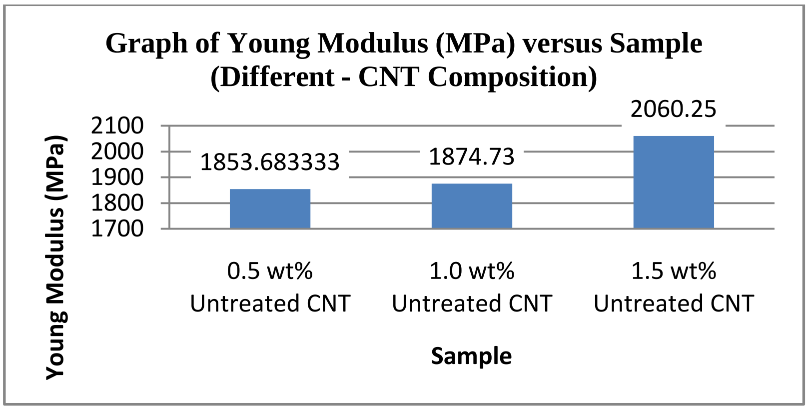


Figure 4.4: Graph of Young Modulus (MPa) versus Sample (Different - CNT Composition)

Graph is showing the Young Modulus Elasticity for sample contain different amount of CNTs which are 0.5wt%, 1.0wt% and 1.5wt%. The highest Young Modulus is by 1.5 wt % of CNTs which are 2060.25 MPa, followed by 1.0 wt % with 1874.73 MPa and the lowest Young Modulus shown by 0.5 wt% with 1853.68 MPa. The data explains that Young Modulus of PLA/CNTs composite is majorly affected by the present of the CNTs.

4.2.3 Elongation (%) Analysis.

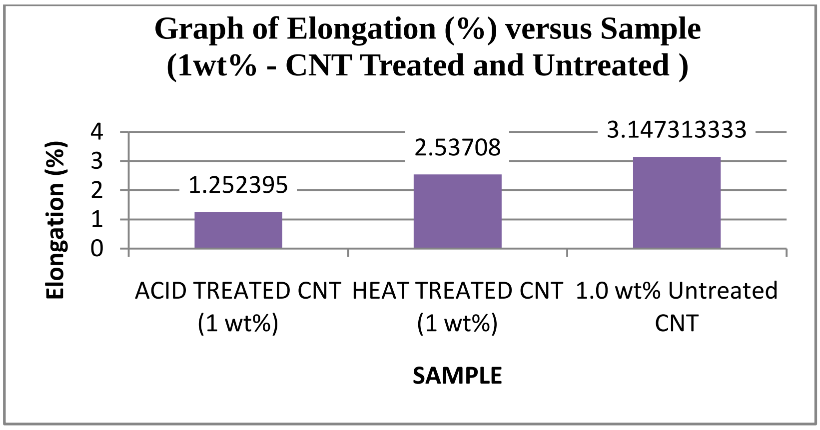


Figure 4.5: Graph of Elongation (%) versus Sample (1wt% - CNT Treated and Untreated)

The figure shows the percentage of elongation obtain by the sample in treated and untreated CNTs in PLA matrix. The highest percentage of elongation is obtain by the untreated sample with 1.00 wt% CNTs with 3.1473%, secondly heat treated CNTs sample with 2.5371% and the smallest elongation obtain by the acid treated CNTs with 1.2524%. Acid treated sample has the smallest elongation due to that the surface of PLA matrix has been effected by the present of small amount of acidic impurities. This also is explained by Defeng Wu et al.(2008) which stated that the thermal degradation properties of PLA/MWCNT treated with acid reduces due to present of impurities such as acidic or basic component because the PLA is an aliphatic ester which will depolimerize when the impurities are presents.

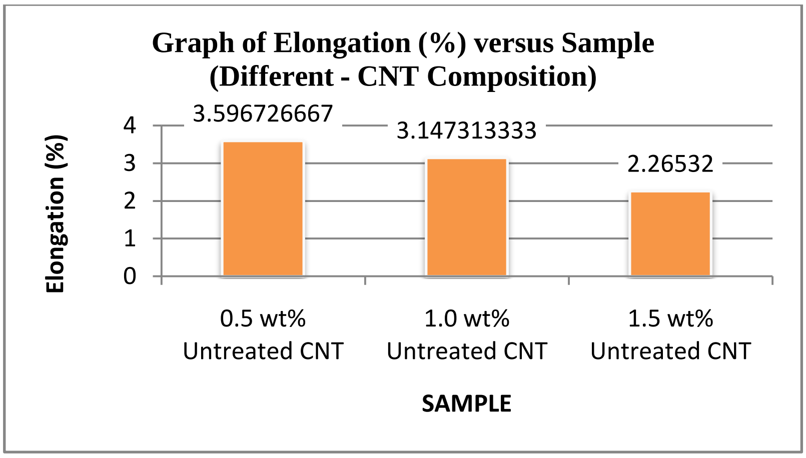


Figure 4.6: Graph of Elongation (%) versus Sample (Different - CNT Composition)

The graph shows the data for elongation of sample for sample in different composition of untreated CNTs which are 0.5%wt, 1.0wt% and 1.5wt%. The data shows that the lower the amount of CNTs the better the elongation of samples. This observation may be explained that incompatibility dispersion of CNTs in polymer matrix.

4.3 SCANNING ELECTRON MICROSCOPE ANALYSIS

4.3.1 Untreated CNTs (1.00wt %) Sample of PLA/CNTs Composite

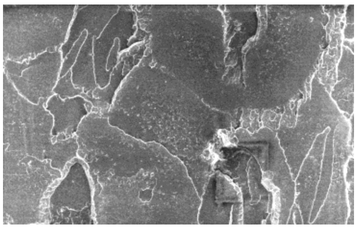


Figure 4.7: 100 times magnification for Untreated CNTs (1.00wt %) Sample

The image shown above is a 100 times magnification using Scanning Electron Microscope (SEM). The image using SEM describe the surface image of sample with 1.00wt % untreated CNTs in PLA matrix which had undergo Universal Testing Machine (UTM) test. The broken or tested sample from UTM testing is analyzed to shows the effect of the testing. The image shows that the surface of the sample is rough and has large chunked of CNTs in PLA matrix.

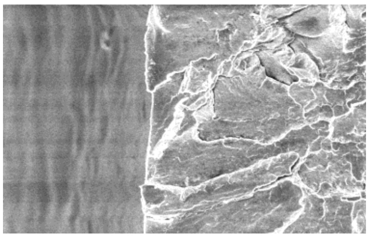


Figure 4.8: 250 times magnification for Untreated CNTs (1.00wt %) Sample

The image shows the further magnification of 1.00wt % untreated CNTs in PLA matrix sample. The surface magnification shows that the surface is rough after undergoing UTM machine testing or tensile tests. The surface image does not give any detail information of the dispersion of CNTs but give the fracture image which is not smooth.

4.3.2 Acid Treated Carbon Nanotube (1.00wt %) Sample of PLA/Carbon Nanotube Composite

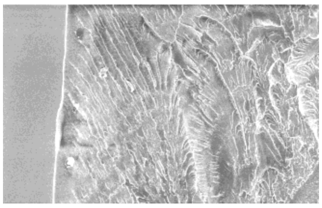


Figure 4.9: 100 times magnification for Acid Treated Carbon Nanotube (1.00wt %) Sample

Image above shows the image of 1.0 wt% CNTs acid treated in PLA matrix. The fracture image shows that the similar observation as 1.0wt% untreated CNTs in PLA matrix which are rough. The fracture image also showing the surface is not smooth after the sample undergo UTM machine testing it also shows that it has uniform type of fracture on the surface.

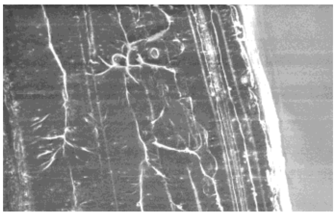


Figure 4.10: 250 times magnification for Acid Treated Carbon Nanotube (1.00wt %) Sample

250 times of magnification of same sample in Figure 4.9 shows that fracture surface is uneven and has some crack line. The surface image also shows that the 1.00wt %CNTs treated with acid has better dispersion compared to of 1.0 wt% CNTs untreated in PLA matrix sample.

4.3.3 Heat Treated Carbon Nanotube (1.00wt %) Sample of Polylactic-acid/Carbon Nanotube Composite

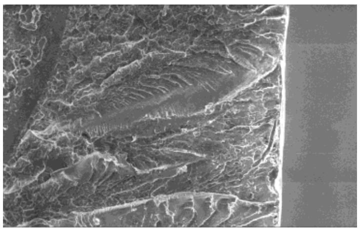


Figure 4.11: 100 times magnification for Heat Treated Carbon Nanotube (1.00wt %) Sample

The image shows the 100 times magnification of fractured sample containing 1.00wt% heat treated CNTs in PLA matrix. The magnification also shows a rough surface after fracture which can be seen from the Figure 4.11 above. The fracture also shows the broken surface has uneven surface.

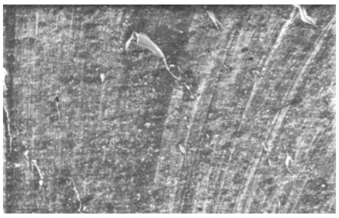


Figure 4.12: 250 times magnification for Heat Treated Carbon Nanotube (1.00wt %) Sample

The closer magnification of 1.00wt% heat treated CNTs in PLA matrix which is 250 times it shows that the dispersion of CNTs in the PLA matrix from the fractured sample is better compared to the 1.0wt% untreated CNTs in PLA matrix or in **Figure 4.8**.

4.4 THERMOGRAVIMETRY ANALYSIS (TGA)

4.4.1 Pure PLA

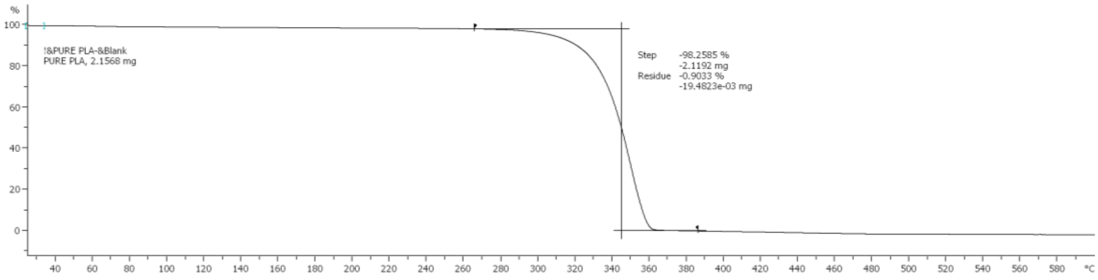


Figure 4.13: Graph of TGA analysis for Percentage of Weight Loss at Different Temperature for Pure PLA

The above image shows the amount of pure PLA mass lost during thermogravimetric analysis. The data shows that the mass loss is around 98.2585% at the range of temperature of 290˚C to 360˚C. Figure above also shows that the residue left from initial is around 0.9033%.

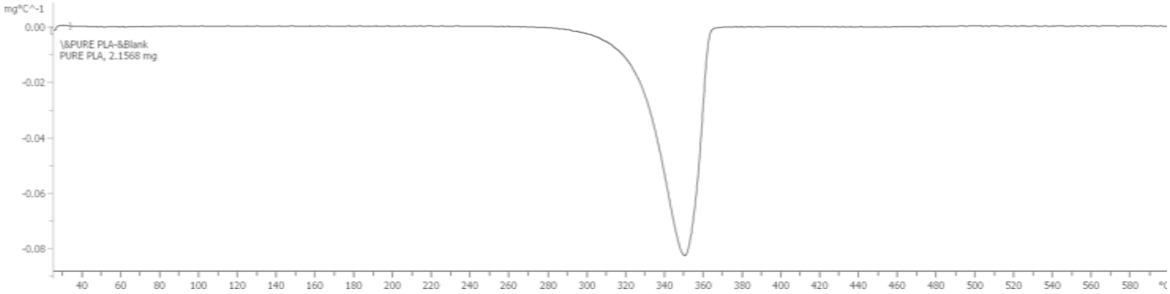


Figure 4.14: Graph of TGA analysis for Thermal Degradation Temperature for Pure PLA

The figure above shows the data for the degradation temperature of Pure PLA. From the graph it shows that the point had the highest degradation temperature is at 350℃. The degradation point for pure PLA at 350 C and this degradation point becomes a guideline for other sample in determining its properties and behavior.

4.4.2 Untreated CNT (0.5 wt %) /PLA composite

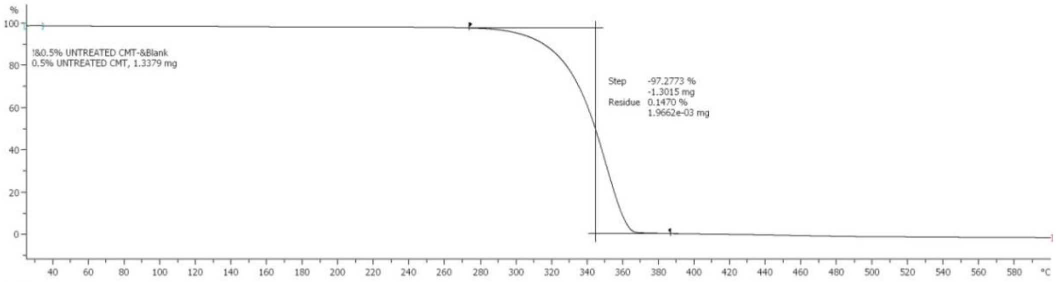


Figure 4.15: Graph of TGA analysis for Percentage of Weight Loss at Different Temperature for Untreated CNT (0.5 wt %) /PLA composite

Based from the graph obtained it shows that the mass of the sample has large reduction of mass the sample reach the temperature range of 300℃ to 370℃. The mass loss during the large drop or degradation point of the sample is around 97.2773 % of mass loss. The y-axis of the graph represent the amount of relative weight loss. The highest gradient or slope from this grpah can be obtained within the temperature range of 330 C to 350 C. S. W. Ko et al. also shown the data of PLA/CNT composite with 2.0

wt% which has the degradation temperature in range of 325 ˚C to 380 ˚C that supported the data which has been obtained. % mass loss at the temperature 325 ˚C.

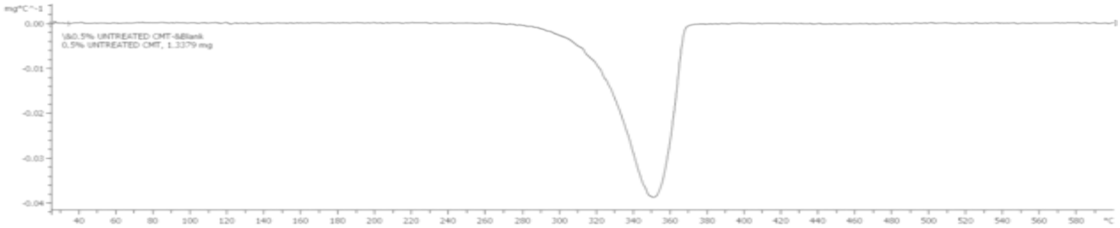


Figure 4.16: Graph of TGA analysis for Thermal Degradation Temperature for Untreated CNT (0.5 wt %) /PLA composite.

From this figure the identification of the exact degradation temperature or the largest amount of relative weight loss occur. So from the graph it can be seen that the degradation tempertaure for 0.5wt% of Carbon Nanotube /PLA composite is at 353 ˚C. This sample which is has higher degradation than virgin PLA shows that adding 0.5wt% CNT untreated has given a slight improvement in the sample ability to degrade at higher temperature. The higher degradation temperature maybe due to the good interaction between the matrix phase (PLA) and fiber phase (CNTs) and goog dispersion.

4.4.3 Untreated Carbon Nanotube (1.0 wt %) /PLA composite

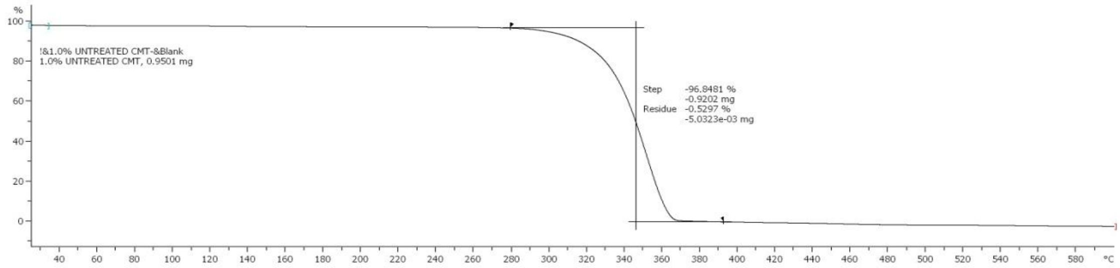


Figure 4.17: Graph of TGA analysis for Percentage of Weight Loss at Different Temperature for Untreated CNT (1.0 wt %) /PLA composite

The graph above shows the mass for sample Untreated CNT (1.0 wt %) /PLA composite. Based from the TGA analysis it shows that the mass loss for the sample is around 96.8481 % and the percentage of residue that left is around 0.5297%. The lower percentage of mass loss shows that the interaction of fiber phase and matrix phase in this sample composition is better than Untreated CNT (0.5 wt %) /PLA composite.

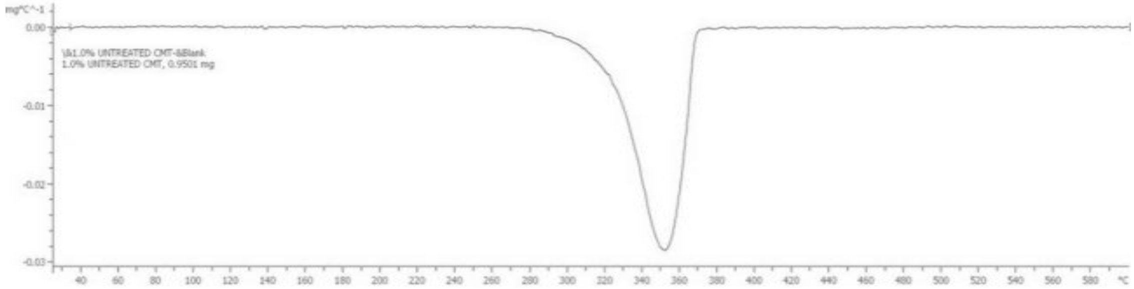


Figure 4.18: Graph of TGA analysis for Thermal Degradation Temperature for Untreated CNT (1.0 wt %) /PLA composite.

The graph above shows the thermal degradation temperature for Untreated CNT (1.0 wt %) /PLA composite. The graph shows that the degradation temperature is high at the range 355 °C for this sample. Higher degradation temperature is a better composite or dispersion of CNTs in the polymer matrix is good. The thermal degradation temperature also shows that the sample is stable at higher temperature before decomposing.

4.4.4 Untreated Carbon Nanotube (1.5 wt %) /PLA composite

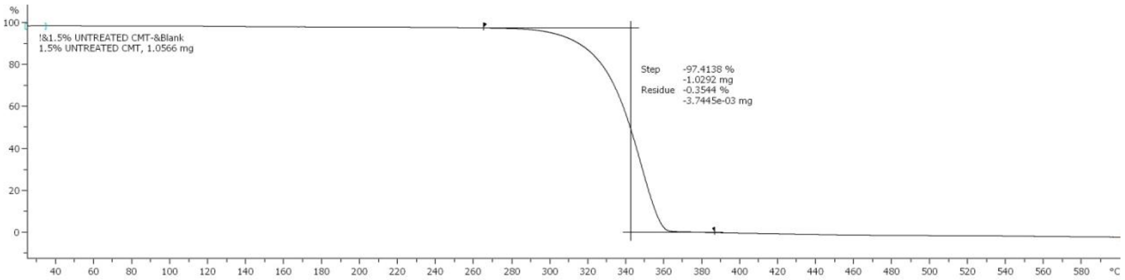


Figure 4.19: Graph of TGA analysis for Percentage of Weight Loss at Different Temperature for Untreated CNT (1.5 wt %) /PLA composite

Graph above shows that amount of mass that loss during a TGA analysis. The mass lost during the degradation point is around 97.4138 % and the residue that left is at 0.3544%. The high percentage mass lost region for this sample is at the region of 320˚C to 360˚ C. The **Figure 4.19** also shows that the amount of sample lost at higher temperature is larger than the pure sample which shows the thermal stability of the sampel at higher temperature.

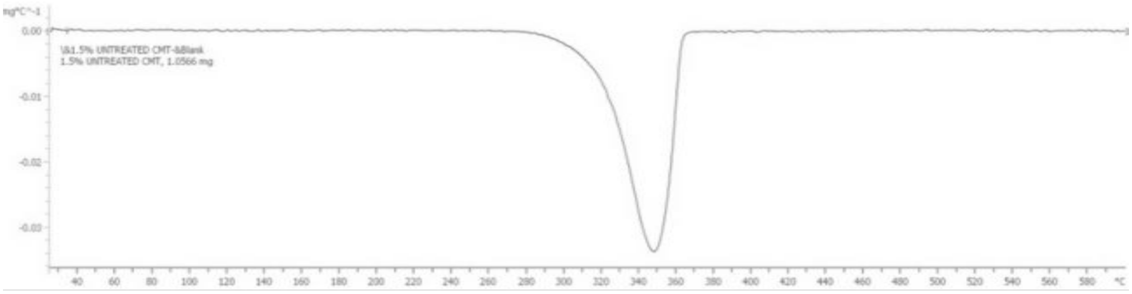


Figure 4.20: Graph of TGA analysis for Thermal Degradation Temperature for Untreated CNTs (1.5 wt %) /PLA composite.

The graph shows the thermal degradation temperature for 1.5 wt% untreated CNTs. From the graph it shows that the value of degradation temperature of the sample is at 348℃. This graph also shows that the sharp drop in the temperature value is within the region of 300˚C to 360˚C. This region where the thermal degradation occur would be the region where the large lost in mass will occur.

4.4.5 PLA/CNTs Composite (1.00wt % Acid Treated Carbon Nanotube)

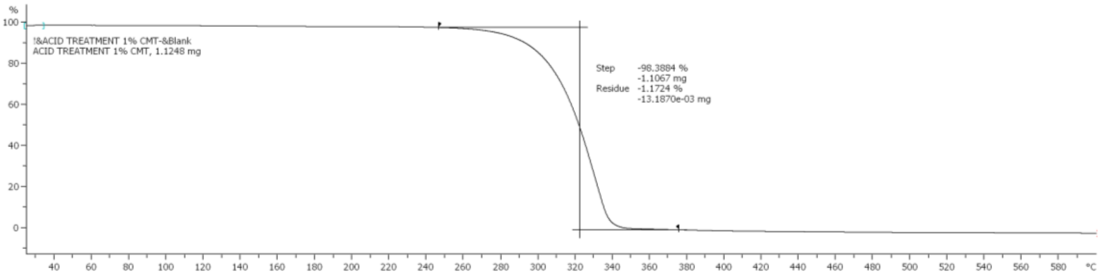


Figure 4.21: Graph of TGA analysis for Percentage of Weight Loss at Different Temperature for Acid Treated CNT (1.0 wt %) /PLA composite

The figure shows the thermogravimetric analysis for PLA/CNT composite for CNTs treated with acid. The sample shows the amount of mass loss is at 98.3884% which at temperature region of 310 C to 330 C. This result sample also shows that the degradation temperature for this sample is much smaller compared to the pure PLA degradation temperature. From, the analytical part it can be conclude that the addition of CNTs which has been treated with acid has causes a decreased in thermal enhancement of PLA which caused it to have smaller thermal degradation temperature. The reason for this error to occur is due to the cause of acid treated CNTs has weaker interaction with the PLA matrix. This is vice versa from the K. Chrissafis (2010) also stated in the paper that the addition of Multi-walled Carbon Nanotube with carboxylic functional group (MWCNTs-COOH) into the polymer matrix has attributed to the improvement of the thermal degradation temperature. Furthermore, a concrete reason for the reduction of thermal degradation temperature is due to PLA is an aliphatic ester which wills depolimerize in the present of acidic or basic impurities from Defeng Wu et al.(2008)

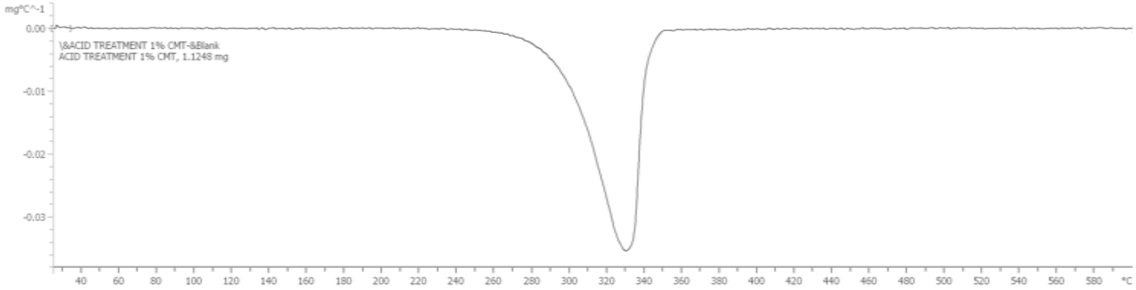


Figure 4.22: Graph of TGA analysis for Thermal Degradation Temperature for Acid Treated CNT (1.0 wt %) /PLA composite.

The figure shows the thermal degradation temperature for this PLA /CNT acid treated sample degrades at the temperature around 330 ° C. The composite has lower thermal degradation temperature due to the dispersion CNTs in the PLA matrix which not constant dispersion and weaker interaction of acid treated CNTs with the polymer matrix. Yoon Jin Kim et al (2005). in the paper written than the MWCNT oxidized under hard condition such as oxidizing with concentrated Nitric acid will cause the crystalline structure are partially damaged. The explanation from Yoon Jin Kim et al.(2005) that weak interaction and dispersion of acid treated CNTs in PLA matrix. Defeng Wu et al.(2008) also stated in the paper that the surface functionalization of MWCNTs also play an important role in the dispersion and thermal stability of PLA matrix. CNTs also could act as inert filler with respect to thermal decomposition of the PLA matrix which has been explained by Defeng Wu et al.(2008), explanation given is that the reduction in cross linking off the matrix chain or on the other hand CNTs act as added component or impurities such as compatibilizer or remaining catalyst from nanotube synthesis may also be considered.

4.4.6 PLA/CNTs Composite (1.00wt % Heat Treated Carbon Nanotube)

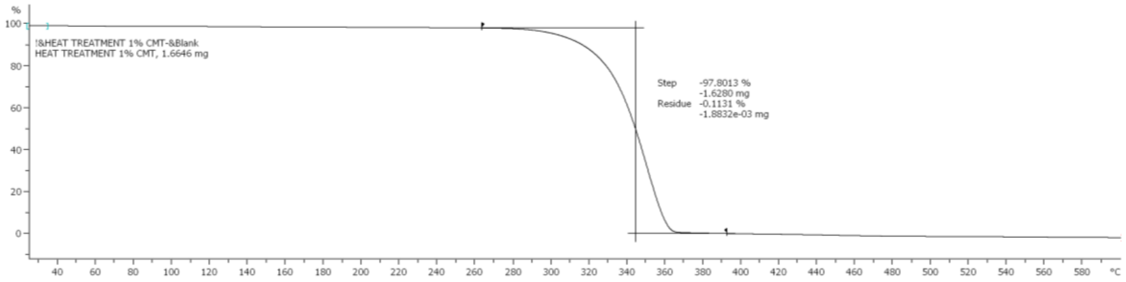


Figure 4.23: Graph of TGA analysis for Percentage of Weight Loss at Different Temperature for Heat Treated CNT (1.0 wt %) /PLA composite

The graph shows the pattern of mass loss in PLA/CNTs composite with 1.00 wt% of heat treated CNTs versus temperature. The graph also shows that the thermal degradation temperature for the composite is higher than 350 C or higher than the pure PLA thermal decomposition temperature. This observation give an indication that heat treated CNTs has better interaction and dispersion in PLA matrix compared to acid treated CNTs.

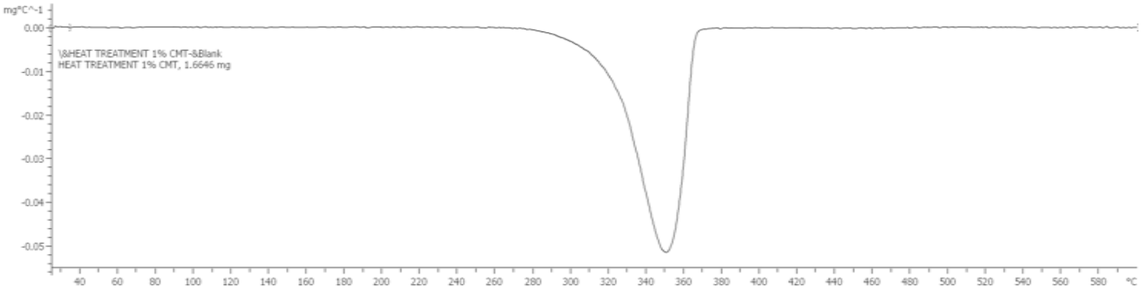


Figure 4.24: Graph of TGA analysis for Thermal Degradation Temperature for Heat Treated CNT (1.0 wt %) /PLA composite.

The figure shows and observation related to the thermal degradation temperature which shows at around 353 C which higher than the pure PLA sample thermal degradation temperature. This observation is an indication that the heat treatment CNTs can give an improvement in thermal degradation property in PLA matrix. Furthmore, this statement is supported by K. Chrissafis (2010) paper which explains the improvement in thermal degradation temperature could be caused by improvement in good matrix-nanotube interaction and good thermal conductivity of the nanotubes and also due to their barrier effect.

4.4.7 Compilation Data for all samples for TGA Analysis

Table 4.1: Compilation Data for TGA analysis

Sample	**Mass Loss (%)**	**Degradation Temperature (̊C)**	**Residue (%)**
Pure PLA	98.2585	350	0.9033
Acid Treated CNTs(1.00wt%)/PLA	98.3884	330	1.1724
Heat Treated CNTs(1.00wt%)/PLA	97.8013	352	0.1131
Untreated CNTs(0.50wt%)/PLA	97.2773	353	0.1470

Untreated CNTs(1.00wt%)/PLA	96.8481	355	0.5297
Untreated CNTs(1.50wt%)/PLA	97.4138	349	0.3544

Table 4.1 shows the data collected from TGA analysis and tabulated by the type of sample used and the percentage mass loss, Degradation Temperature and Residue collected. The data is retrieved from the graph of the entire sample to find the comparison between different samples used. To find a better pattern of comparison a bar chart is plotted in Figure 4.25 and 4.26 to give an overall view and trend that occurs in different sample.

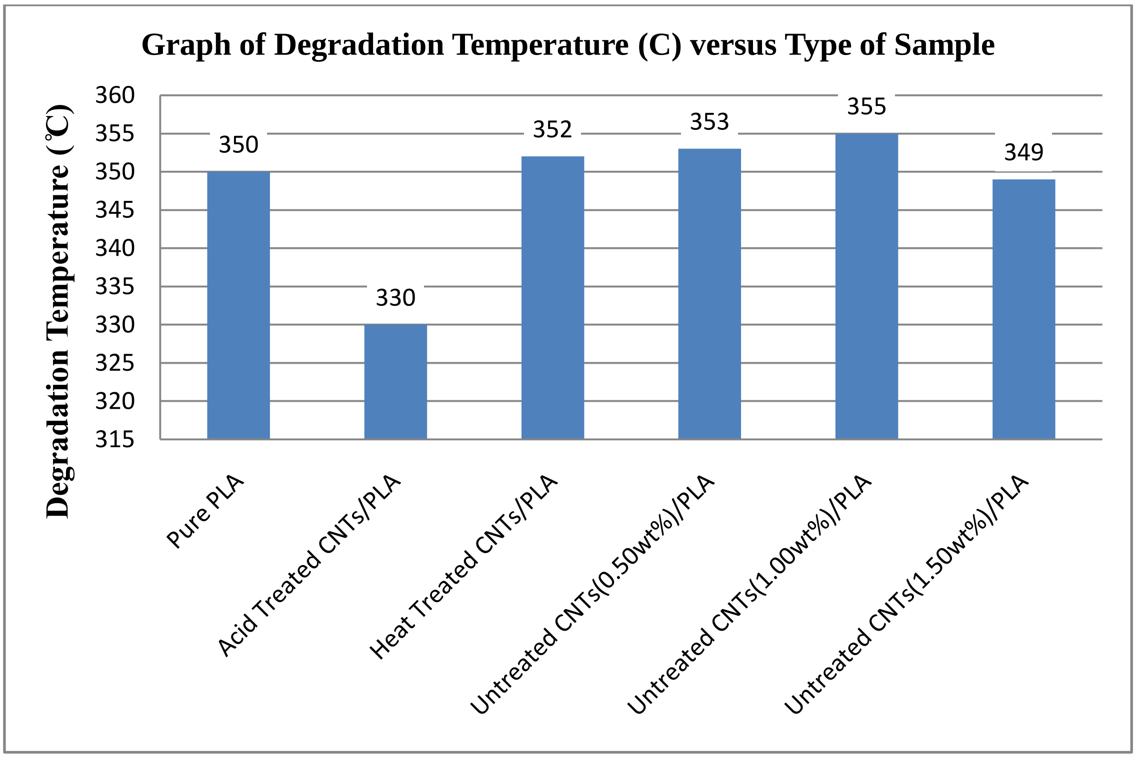


Figure 4.25: Graph of Degradation Temperature (C) versus Type of Sample

The figure above shows the degradation temperature for different sample. The highest degradation temperature shown by the 1.00wt % of Untreated CNT/PLA composite, than 0.50wt % of Untreated CNT/PLA composite, Heat treated (1.00wt% CNTs), Pure PLA, 1.50wt % of Untreated CNT/PLA composite and the lowest degradation temperature shown by the acid treated sample. This pattern shows that inconsistency of degradation temperature when the amount of Untreated CNTs increases the reason for this pattern is due to the inconsistency of untreated CNTs dispersion in the sample. Furthermore, the higher dispersion or higher consistency of CNTs will improve the degradation temperature imputable of CNTs properties as excellent heat conductor.

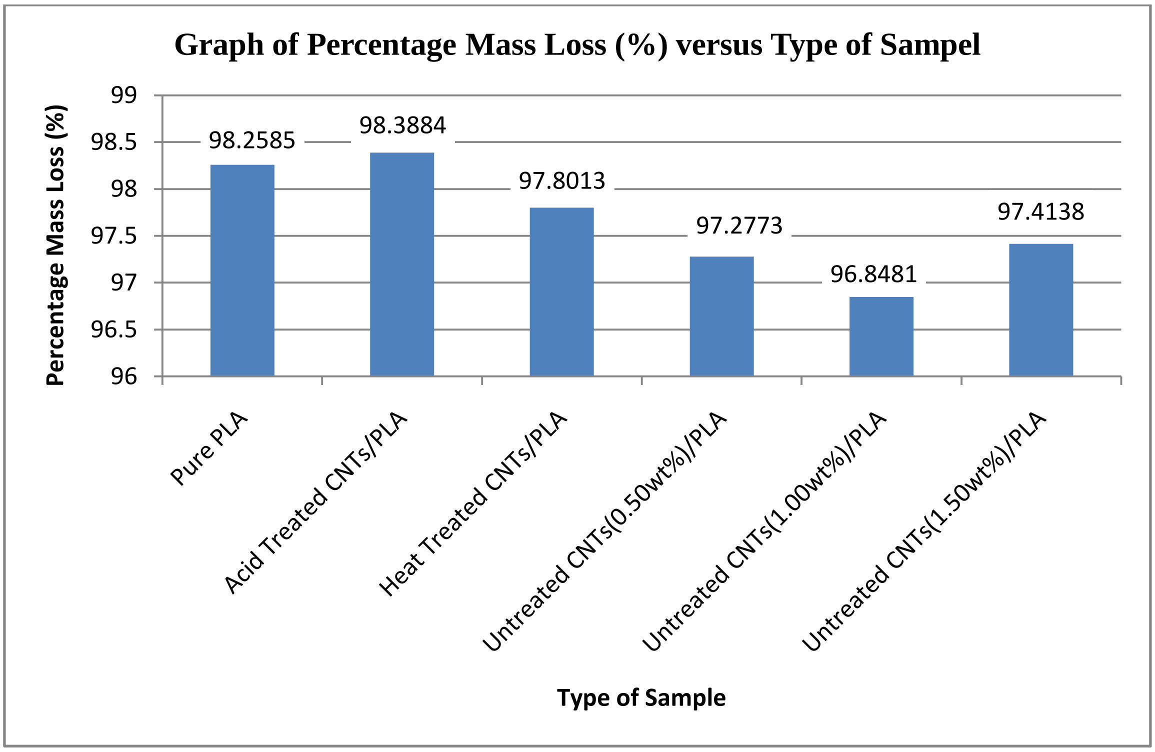


Figure 4.26: Graph of Percentage Mass Loss (%) versus Type of Sample

The figure above shows the percentage mass loss when different sample are compared. The trend of percentage mass loss followed by the highest which is acid treated sample, secondly Pure PLA, thirdly treated (1.00wt% CNTs), fourthly 1.50wt % of Untreated CNT/PLA composite, fifthly 0.50wt % of Untreated CNT/PLA composite and finally the lowest percentage mass loss is found from the 1.00wt % of Untreated CNT/PLA composite. This pattern shows the trend of the consistent dispersion in mass play a major role in the amount of mass loss during TGA analysis because the sample with good dispersion of CNTs will have smaller mass loss compared to sample having moderate or low dispersion of CNTs this is supported by the good mechanical and thermal properties that exhibit by the CNTs.

4.5 PLA/CNT COMPOSITE SURFACE RESISTIVITY

4.5.1 Surface Resistivity (Ohm/sq) versus Sample with 1.00 wt% CNTs (Treated and Untreated Sample)

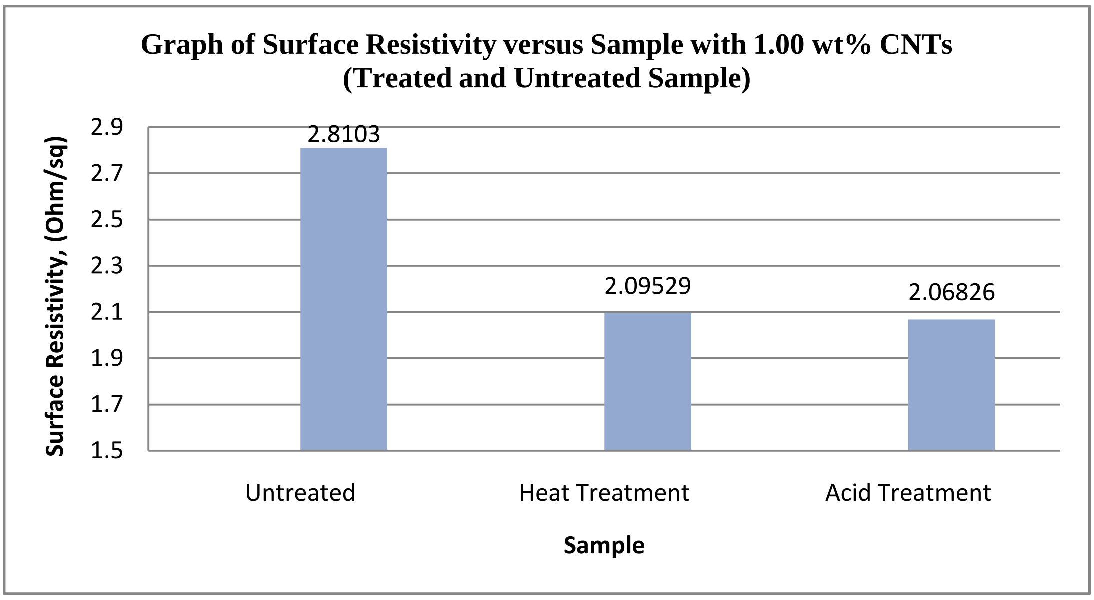


Figure 4.27: Graph of Surface Resistivity versus Sample with 1.00 wt% CNTs (Treated and Untreated Sample)

The data above shows the surface resistance in different sample which are untreated CNTs, acid treated CNTs and heat treated CNTs. Based on data obtain above it shows that the surface resistivity of Acid treated CNTs has lowest surface resistivity compared to heat treated and Untreated CNTs sample. The percentage different in surface resistivity between acid treated CNTs sample and untreated CNTs is 26.40 % and the percentage different in surface resistivity between acid treated CNTs sample and heat treated CNTs samples is lower which is 1.29 %. The data obtain also shows that acid treated sample according to Chen-Feng Kuana et al.(2008) has surface resistivity compared to untreated CNTs sample.

4.5.2 Surface Resistivity (Ohm/sq) versus Sample with Different Composition of CNTs

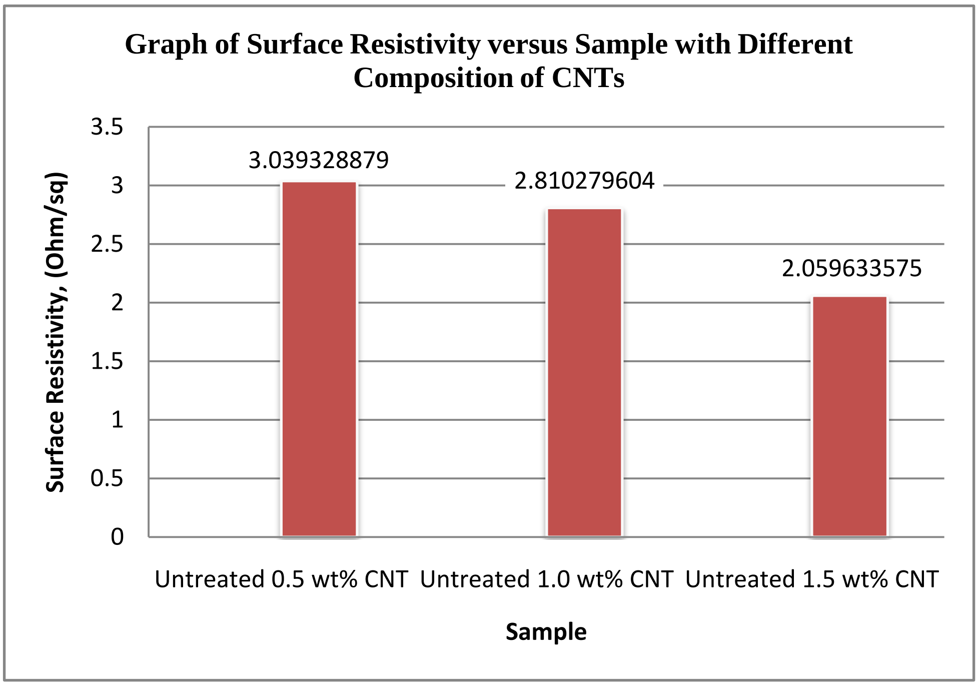


Figure 4.28: Graph of Surface Resistivity versus Sample with Different Composition of CNTs

The data obtain for untreated sample In different composition of CNTs which are 0.5wt%, 1.0 wt% and 1.5wt%. The surface resistivity shown explains that the lower the amount of CNTs the higher the conductivity which is vice versa from the explanation done from Chen-Feng Kuana et al.(2008) which shows the higher the amount CNTs the higher the conductivity. This observation of surface resistivity and CNTs composition is due to the inconsistent dispersion of CNTs in the sample with untreated CNTs. The observation also can be explained based from Chen-Feng Kuana et al.(2008) is that the paper explain that the surface resistivity reduces as the amount CNTs increasing is because the paper uses acid treated MWCNT and not untreated CNTs which produces different data and results.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

As a conclusion this data obtain from Mechanical testing, SEM, TGA and surface resistant shows a variation of results independent of sample. From the mechanical testing using UTM machine the result for untreated sample is that higher the weight percentage of CNTs will produce higher Young Modulus but the data for elongation shows a vice versa results and tensile strength data obtain for this sample shows inconsistency of tensile strength with the weight percent of untreated CNTs. Meanwhile, for mechanical testing for untreated and treated sample with 1.00wt % CNTs shows that the acid and heat treatment done has produce weaker or less strong sample compared to sample contain untreated CNTs.

SEM analysis done for treated and untreated sample with same CNTs weight percentage shows the fracture structure that has been obtained. The analysis shows that s fracture for the entire sample produces are rough surface and not a clean fracture.

Meanwhile, from TGA analysis the data obtain shows that acid treatment will produce lower degradation temperature sample due to present of acidic or basic impurities from the sample. The data also shows that the highest degradation temperature is obtained from the sample contain 1.0wt% of untreated sample.

Surface Resistivity analysis shows that the acid treatment sample will produce a lower surface resistivity sample compared to heat treated and untreated sample. On the other hasnd, the data obtain for sample containg different weight percentage of untreated CNTs shows that the surface resistivity increases as the higher the weight percentage of untreated CNTs.

5.2 RECOMMENDATION

In improving this study in future several recommendations can be made such as:-

i) By making more variation in the amount of untreated CNTs in weight percent such as 2.0wt % to 5.0wt % to identify the effect of untreated CNTs in PLA matrix.
ii) Improving the parameter controlling acid treatment process. Such as the time for the reaction of CNTs and acid, the concentration of acid used the amount of acid used and the heating temperature of the acid CNTs mixture.
iii) By improving the heating treatment process by making variation in the temperature range.
iv) Improvement in the weight percentage of CNTs for treated sample.

This improvement can be made to ensure that future study of this production of conductive polymer can be made effecting and applicable in a larger scale.

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APPENDIX A

SAFETY (MSDS) FOR CARBON NANOTUBE

1. CHEMICAL PRODUCT

Product name : Ctube-100, Ctube-101, Ctube-120
Chemical name : Multiwall Carbon Nanotubes
Formula : Carbon
Chemical family : Synthetic Graphite
Synonyms : Multi-Walled Carbon Nanotube

2. COMPOSITION / INFORMATION ON INGREDIENTS

COMPONENT	%	O/D	LENGTH	CAS NO.
Carbon Nanotube Synthetic graphite	95 MIN.	10~40nm	1~25□ m	7782-42-5

3. HAZARDS IDENTIFICATION

Irritant Irritating to eyes and respiratory system
HMIS Rating
Health 2
Flammability 0
Reactivity 0
NFPA Rating
Health 2
Flammability 0
Reactivity 0

4. FIRST AID MEASURES

a) Oral Exposure - if swallowed, wash out a mouth with copious amount of water provided person is conscious. Call a physician

b) Inhalation exposure - if inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen
c) Dermal exposure - immediately wash affected area with soap and copious amounts of water
d) Eye exposure - immediately flush eyes with copious amounts of water for at least 15 minutes

5. FIRE FIGHTING MEASURES

a) Flash : Not available
b) Explosion limits : Unknown
c) Extinguishing media : Water, Carbon Dioxide, Dry chemical or Foam
d) Decomposition products : Carbon Monoxide, Carbon Dioxide
e) Unusual hazards : Thermal decomposition or combustion may produce dense smoke

6. ACCIDENTAL RELEASE MEASURES

a) **Procedure of personal precaution** wear respirator, chemical safety goggles, rubber boots to avoid skin contact
b) Methods for cleaning up sweep up, place in a bag and hold for waste disposal, avoid raising dust. Ventilate area and wash spilt site after material pickup is completed

7. HANDLING AND STORAGE

a) User Exposure - Do not breathes dust. Avoid contact with eyes, skin and clothing Avoid prolonged or repeated exposure

b) Storage Suitable - Keep tightly closed and store in a cool & dry place

8. EXPOSURE CONTROLS / PERSONAL PROTECTION

a) **EYE PROTECTION** Use approved safety glasses or goggles

b) **RESPIRATOR** Wear high-efficiency dust mask

c) **SKIN PROTECTION** Impervious gloves and protective clothing to prevent skin contact

d) VENTILATION A local or general exhaust system is recommended

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance : Clumpy, Black Or Dark Gray Powder
Molecular weight : n/a
Boiling point : n/a
Melting point : 3,652~3,697˚C
Vapor pressure : : N/A
Freezing point : N/A
Specific gravity : 2.1g/cm3 at 25˚C
Bulk density : 0.03~0.06g/cm3
Volatile : % N/A
Water content : N/A
Solvent content : N/A
Solubility in water : Insoluble
Ph : 6-7
Odor : Odorless
N/A = Not Available

10. STABILITY AND REACTIVITY

a) Stability : Stable

b) Hazardous Polymerization : None

c) Hazardous Decomposition Products: Carbon monoxide, Carbon Dioxide

d) Materials To Avoid : Strong oxidizing agents

11. TOXICOLOGICAL INFORMATION

Route of Exposure

Skin : Contact May cause skin irritation
Eye Contact : Cause eye irritation

Inhalation : Material is irritating to mucous membranes and upper respiratory tract

Ingestion : May be harmful if swallowed

12. ECOLOGICAL INFORMATION

No known ecological harms

13. DISPOSAL CONSIDERATIONS

Disposal - Dispose of this material in accordance with local, state, and federal regulation

14. TRANSPORTATION INFORMATION

DOT

Proper Shipping Name None

Non–Hazardous For Transport This substance is considered to be non – hazardous for transport.

IATA

Non–Hazardous For Air Transport This substance is Non – hazardous for air transport

APPENDIX B

SAFETY (MSDS) FOR POLYLACTIC ACID (PLA)

1. CHEMICAL PRODUCT

Product name : MiniFIBERS PolyLactic Acid Fibers

Synonyms : PLA, Polylactide, Aliphatic Polyester

2. COMPOSITION / INFORMATION ON INGREDIENTS

COMPONENT	%	CAS NO.
PolyLactic Acid	>99%	26100-51-6

3. HAZARDS IDENTIFICATION

- This product is not hazardous under the criteria of U.S. Occupational Safety and Health Standard 29 CFR 1910 Subpart Z and United Nations GHS Parts 2, 3, and 4.

4. FIRST AID MEASURES

a) Inhalation: Inhalation of fibers or fiber dust may cause respiratory irritation. Move to fresh air if effects occur. Consult a physician if persistent coughing or other symptoms develop.

b) Skin: If thermal burn, cool with water and seek immediate medical attention; do not attempt to peel fibers from skin. Non-thermal contact with fibers may cause mechanical irritation of the skin. Wash off with soap and water, and consult a physician if symptoms develop.

c) Skin Absorption: A single prolonged skin exposure is not likely to result in the material being absorbed through the skin in harmful amounts.

d) Eyes: Fibers or fiber dust may cause irritation or scratch the surface of the eyes. Flush with water to remove particles; remove contact lenses if present part eyelids with fingers to ensure complete flushing. Consult a physician if persistent irritation or other symptoms develop.

e) Ingestion: No adverse effects are believed to occur from swallowing a small amount. Consult a physician if symptoms develop or if a large amount is swallowed.

*Contaminated clothing does not need to be removed.

*Personal protective equipment is not required for first-aid responders

5. FIRE FIGHTING MEASURES

a) Flash Point: 121oC
b) Flammable Limits: Not determined
c) Extinguishing Media: Water, dry chemicals.
d) Hazardous Combustion Products: May include, but are not limited to, CO and CO2.
e) Unusual Fire & Explosion Hazards: None known.
f) Special Fire Fighting Procedures: Avoid excessive inhalation of smoke or potential thermal decomposition products. Keep product cool by spraying with water. If outdoors, fight fire from an upwind position.
g) Special Protective Equipment: Due to potential decomposition of the polymer, firefighters should be equipped with positive pressure self-contained breathing apparatus (SCBA) and standard protective fire fighting clothing (helmet, eye protection, overalls, boots, and gloves) when fighting all indoor fires and any significant outdoor fires.

6. ACCIDENTAL RELEASE MEASURES

a) Personal Precautions: None needed.
b) Environmental Precautions: None needed.
c) Methods for Cleanup: Vacuum or sweep up and place in a standard disposal container. Avoid the use of air jets.

7. HANDLING AND STORAGE

a) Precautions for Safe Handling: No special handling has been shown to be necessary.
b) Conditions for Safe Storage: Avoid overstacking to prevent collapse or breakage of the package . Do not store near flame , oxidizing materials, or incompatible substances. Store at temperatures below 60°C (150 ° F).
c) Other Precautions: None

8. EXPOSURE CONTROLS / PERSONAL PROTECTION

a) Exposure Guidelines: Fiber dust should be considered a nuisance dust, i.e. particulates (not otherwise classified): ACGIH Threshold Limit Value: 10 mg/m3 total dust; 3-mg/m3 respirable dust OSHA Permissible Exposure Limit: 15 mg/m3 total dust; 5-mg/m3 respirable dust
b) Engineering Controls: Local exhaust recommended to reduce exposure to fiber dust.
c) Specific Personal Protective Equipment:
d) Respiratory: For operations where inhalation exposure can occur, a NIOSH approved dust mask/respirator is recommended.
e) Eye: For operations where eye contact can occur, eye protection such as goggles or safety glasses is recommended.
f) Hand: Protective gloves not required.
g) Skin/Other: Not required.
h) Work/Hygienic Practices: Maintain good housekeeping to control dust accumulations. Avoid the use of air jets to blow off equipment; use vacuums instead.

9. PHYSICAL AND CHEMICAL PROPERTIES

a) Chemical Formula: -(C6H8O5)-
b) Flash Point: 121oC
c) Solubility: Not soluble
d) Appearance: White solid

e) Evaporation Rate: Does not apply
f) Partition Coefficient: No data available
g) Odor: No odor
h) Flammability: Non-flammable
i) Auto-Ignition Temp: No data available
j) pH: No data available
k) Vapor Pressure: Does not apply
l) Decomposition Temp:No data available
m) Melting Point: 130-170oC
n) Vapor Density: Does not apply
o) Viscosity: Does not apply
p) Boiling Point: Does not apply
q) Specific Gravity: 1.23-1.30 g/cm3

10. STABILITY AND REACTIVITY

a) Reactivity: Data not available.
b) Stability: Stable under normal conditions.
c) Hazardous Polymerization: Will not occur.
d) Conditions to Avoid: Excessive heat should be avoided.
e) Incompatible Materials: Tends to decompose in strong acids and bases.
f) Hazardous Decomposition Products: None anticipated under normal or recommended handling and storage conditions

11. TOXICOLOGICAL INFORMATION

a) Health Hazards (Acute and Chronic): No data available.
b) Carcinogenicity:
c) NTP: Not listed.
d) IARC: 3 - Not classifiable as to its carcinogenicity to humans.
e) OSHA: Not regulated.
f) Signs and Symptoms of Exposure: No data available.

g) Medical Conditions Aggravated by Exposure: Some individuals, e.g. with asthma or bronchitis, are likely to be intolerant of high concentrations of airborne fibers or fiber dust when processing

12. ECOLOGICAL INFORMATION

a) Toxicity: No data available.
b) Persistence and Degradability: No data available. Fiber is biodegradable.
c) Bioaccumulative Potential: No data available.
d) Mobility in Soil: No data available.

13. DISPOSAL CONSIDERATIONS

Waste Disposal Method**:** Dispose of in accordance with all applicable governmental regulations for non-hazardous solid waste. May be recycled, composted, incinerated, or landfilled. Recycling of corrugated packaging is encouraged where possible. Other packaging may be disposed of with product. Standard disposal containers are acceptable.

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14. TRANSPORTATION INFORMATION

- Proper Shipping Name: Polyester Staple Fiber
- U.S. DOT: Not regulated.
- U.S. NMFC Item Number: 68310
- ICAO/IATA: Not regulated.
- HTC Number: 5503.20
- IMDG: Not regulated.
- U.N. Number: None
- Canada TDG: Not regulated.

APPENDIX C

SAFETY (MSDS) FOR NITRIC ACID

1. CHEMICAL PRODUCT

Product Name: Nitric acid, 65%

Catalog Codes: SLN2161

CAS#: Mixture.

RTECS: Not applicable.

TSCA: TSCA 8(b) inventory: Water; Nitric acid, fuming

CI#: Not applicable.

Synonym: Nitric Acid, 65%

Chemical Name: Not applicable.

Chemical Formula: Not applicable.

2. COMPOSITION / INFORMATION ON INGREDIENTS

Name	CAS #	% by Weight
Water	7732-18-5 35	35
Nitric acid,	7697-37-2 65	65

3. HAZARDS IDENTIFICATION

a) Potential Acute Health Effects:

- Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion,. Slightly hazardous in case of inhalation (lung sensitizer). Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Prolonged exposure may result in skin burns and ulcerations. Over-exposure by inhalation may cause respiratory irritation. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

b) Potential Chronic Health Effects:
  - CARCINOGENIC EFFECTS: Not available. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to lungs, mucous membranes, upper respiratory tract, skin, eyes, and teeth. Repeated or prolonged exposure to the substance can produce target organs damage. Repeated or prolonged contact with spray mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to spray mist may produce respiratory tract irritation leading to frequent attacks of bronchial infection.

4. FIRST AID MEASURES

a) Eye Contact:
  - Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

b) Skin Contact:
  - In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothingand shoes. Cover the irritated skin with an emollient. Cold water may be used.Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

c) Serious Skin Contact:
  - Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

d) Inhalation:
   - If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

e) Serious Inhalation:
   - Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek immediate medical attention.

f) Ingestion:
   - If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

g) Serious Ingestion: Not available.

5. FIRE FIGHTING MEASURES

a) Flammability of the Product: Non-flammable.
b) Auto-Ignition Temperature: Not applicable.
c) Flash Points: Not applicable.
d) Flammable Limits: Not applicable.
e) Products of Combustion: Not available.
f) Fire Hazards in Presence of Various Substances: of combustible materials
g) Explosion Hazards in Presence of Various Substances: Explosive in presence of reducing materials, of organic materials, of metals, of alkalis. Non-explosive in presence of open flames and sparks, of shocks.
h) Fire Fighting Media and Instructions: Not applicable.
i) Special Remarks on Fire Hazards: Flammable in presence of cellulose or other combustible materials. Phosphine, hydrogen sulfide, selenide all ignite when fuming nitric acid is dripped into gas. (Nitric Acid, fuming)

j) Special Remarks on Explosion Hazards: Reacts exlposively with metallic powders, carbides, cyanides, sulfides, alkalies and turpentine. Can react explosively with many reducing agents. Arsine, phosphine, tetraborane all oxidized explosively in presence of nitric acid. Cesium and rubidiumacetylides explode in contact with nitric acid. Explosive reaction with Nitric Acid + Nitrobenzene + water. Detonation with Nitric Acid + 4-Methylcyclohexane. (Nitric acid, fuming)

6. ACCIDENTAL RELEASE MEASURES

a) Small Spill:
   - Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container. If necessary: Neutralize the residue with a dilute solution of sodium carbonate.

b) Large Spill:
   - Corrosive liquid. Oxidizing material. Poisonous liquid. Stop leak if without risk. Absorb with DRY earth, sand or other noncombustible material. Do not get water inside container. Avoid contact with a combustible material (wood, paper, oil, clothing.). Keeps substance damp using water spray. Do not touch spilled material. Use water spray curtain to divert vapor drift. Use water spray to reduce vapors. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Neutralize the residue with a dilute solution of sodium carbonate. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

7. HANDLING AND STORAGE

a) Precautions:
   - Keep locked up.. Keep container dry. Keep away from heat. Keep away from sources of ignition. Keep away from combustible material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Never add water to this product. In case

of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as reducing agents, combustible materials, organic materials, metals, acids, alkalis, moisture. May corrode metallic surfaces. Store in a metallic or coated fiberboard drum using a strong polyethylene inner package.

b) Storage:

- Keep container tightly closed. Keep container in a cool, well-ventilated area. Separate from acids, alkalies, reducing agents and combustibles. See NFPA 43A, Code for the Storage of Liquid and Solid Oxidizers. Do not store above 23°C (73.4°F).

8. EXPOSURE CONTROLS / PERSONAL PROTECTION

a) Engineering Controls:

Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

b) Personal Protection:

Face shield. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves. Boots.

c) Personal Protection in Case of a Large Spill:

Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

d) Exposure Limits: TWA: 2 STEL: 4 (ppm) from ACGIH (TLV)

[United States] TWA: 2 STEL: 4 from OSHA (PEL) [United States] Consult local authorities for acceptable exposure limits.

9. PHYSICAL AND CHEMICAL PROPERTIES

a) Physical state and appearance: Liquid.

b) Odor: Acrid. Disagreeable and choking. (Strong.)
c) Taste: Not available.
d) Molecular Weight: Not applicable.
e) Color: Colorless to light yellow.
f) pH (1% soln/water): Acidic.
g) Boiling Point: 121°C (249.8°F)
h) Melting Point: -41.6°C (-42.9°F)
i) Critical Temperature: Not available.
j) Specific Gravity: 1.408 (Water = 1)
k) Vapor Pressure: 6 kPa (@ 20°C)
l) Vapor Density: 2.5 (Air = 1)
m) Volatility: Not available.
n) Odor Threshold: 0.29 ppm
o) Water/Oil Dist. Coeff.: Not available.
p) Ionicity (in Water): Not available.
q) Dispersion Properties: See solubility in water, diethyl ether.
r) Solubility:Easily soluble in cold water, hot water. Soluble in diethyl ether.

10. STABILITY AND REACTIVITY

a) Stability: The product is stable.
b) Instability Temperature: Not available.
c) Conditions of Instability: Incompatible materials
d) Incompatibility with various substances: Highly reactive with alkalis. Reactive with reducing agents, combustible materials, organic materials, metals, acids.
e) Corrosivity: Extremely corrosive in presence of aluminum, of copper. Non-corrosive in presence of glass, of stainless steel(304), of stainless steel(316), of brass.
f) Special Remarks on Reactivity: A strong oxidizer. Reacts violently with alcohol, organic material, turpene, charcoal. Violent reaction with Nitric acid + Acetone and Sulfuric acid. Nitric Acid will react with water or steam to produce heat and toxic, corrosive and flammable vapors. (Nitric acid, fuming)

g) Special Remarks on Corrosivity: In presence of traces of oxides, it attacks all base metals except aluminum and special chromium steels. It will attack some forms of plastics, rubber, and coatings. No corrosive effect on bronze. No corrosivity data for zinc, and steel
h) Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

a) Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.
b) Toxicity to Animals: LD50: Not available. LC50: Not available.
c) Chronic Effects on Humans: Contains material which may cause damage to the following organs: lungs, mucous membranes, upper respiratory tract, skin, eyes, teeth.
d) Other Toxic Effects on Humans: Extremely hazardous in case of inhalation (lung corrosive). Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (corrosive), of ingestion, Special Remarks on Toxicity to Animals: LDL - Lowest Published Lethal Dose [Human] - Route: Oral; Dose: 430 mg/kg (Nitric acid, fuming)
e) Special Remarks on Chronic Effects on Humans: May cause adverse reproductive effects (effects on newborn and fetotoxicity) based on animal data. (Nitric acid, fuming)
f) Special Remarks on other Toxic Effects on Humans: Acute Potential Health Effects: Skin: Severely irritates skin. Causes skin burns and may cause deep and penetrating ulcers of the skin with a characteristic yellow to brownish discoloration. May be fatal if absorbed through skin.
g) Eyes: Severely irritates eyes. Causes eye burns. May cause irreversible eye injury. Ingestion: May be fatal if swallowed. Causes serious gastrointestinal tract irritation or burns with nausea, vomiting, severe abdominal pain, and possible "coffee grounds" appearance of the vomitus . May cause perforation of the digestive tract.
h) Inhalation: May be fatal if inhaled. Vapor is extremely hazardous. Vapor may cause nitrous gas poisoning. Effects may be delayed. May cause irritation of the

mucous membranes and respiratory tract with burning pain in the nose and throat, coughing, sneezing, wheezing, shortness of breath and pulmonary edema. Other symptoms may include nausea, and vomiting. Chronic Potential Health Effects: Repeated inhalation may produce changes in pulmonary function and/or chronic bronchitis. It may also affect behavior (headache, dizziness, drowsiness, muscle contaction or spasticity, weakness, loss of coordinaton, mental confusion), and urinary system (kidney faillure, decreased urinary output after several hours.

12. **ECOLOGICAL INFORMATION**

a) Ecotoxicity: Not available.
b) BOD5 and COD: Not available.
c) Products of Biodegradation: Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.
d) Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.
e) Special Remarks on the Products of Biodegradation: Not available.

13. **DISPOSAL CONSIDERATIONS**

Waste Disposal:

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

14. **TRANSPORTATION INFORMATION**

- DOT Classification: Class 8: Corrosive material
- Identification: : Nitric acid UNNA: 2031 PG: II
- Special Provisions for Transport: Marine Pollutant