PRODUCTION & CHARACTERIZATION OF MULTI-WALLED CARBON NANOTUBE (CARBOXYL -FUNCTIONALIZED) REINFORCED POLYESTERS

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Thesis submitted in partial fulfilment of the requirements

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SUPERVISOR’S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering (Hons).

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Dedication

Special dedication to my beloved mother, Mrs. Sarojini Jeganmohan, my supervisor, friends, my fellow colleague and all faculty members for all your care, support and believe in me.

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I would like to thank God, for giving me good strength and health to do my PSM. Without Him, nothing is possible and I wouldn’t be able to do my PSM work.

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I acknowledge my sincere indebtedness and gratitude to my parents for their love, dream and sacrifice throughout my life. They always support me to go for further studies and have faith on me to fulfil my studies. I cannot find the appropriate words that could properly describe my appreciation to them.

ABSTRACT

This paper presents the study on the production and characterization of Carbooxyl functionalized Multi-Walled Carbon Nanotube (MWCNT) reinforced Polyester composite. The main objective of this study is to optimize the weight percentage of MWCNT in the composite and to optimize the sonication time of the composite. To determine the optimum weight percentage of MWCNT, samples with different weight percentage of MWCNT was prepared. The prepared samples were sonicated for one hour and were tested for its viscosity. From the result, the viscosity was the highest at 0.4wt % MWCNT. Hence, the optimum weight percentage of MWCNT is at 0.4wt % MWCNT. After optimizing the weight percentage of MWCNT, the sonication time was optimized. This was done by preparing samples of 0.4wt % MWCNT which was then sonicated at different time intervals. After sonication, the viscosity was determined. From the result, the viscosity was the highest for the sample that was sonicated for 90 minutes. Hence, the optimum sonication time of 0.4wt % MWCNT is 90 minutes. After optimization, the composite was then characterized using Tensile Strength (TS) test, Thermal Gravimetric Analysis (TGA) and Fourier Transform Infrared Spectroscopy (FTIR). From the Tensile Strength test, it can be concluded that the composite with 0.4wt % MWCNT was stronger than the sample without MWCNT. Due to its high mechanical strength and other unique properties, this Polyester composite has the potential to be used in various applications.

Keywords: Polyester resin; Carbon nanotube; Viscosity; Dispersion; Ultrasonication

ABSTRAK

Kertas kerja ini membentangkan kajian mengenai pengeluaran dan pencirian komposit Poliester yang diperkukuhkan dengan Tiub Nano Karbon “Multi Walled” fungsi Karboksil (MWCNT). Objektif utama kajian ini adalah untuk mengoptimumkan peratusan berat MWCNT dalam komposit dan masa sonikasi komposit. Untuk menentukan peratusan berat optimum MWCNT, sampel dengan peratusan berat badan MWCNT yang berbeza telah disediakan. Sampel yang disediakan telah disonikasi selama satu jam dan diuji untuk kelikatan. Dari keputusan itu, kelikatan adalah yang tertinggi di 0.4wt % MWCNT. Oleh itu, peratusan berat optimum MWCNT pada 0.4wt % MWCNT. Selepas mengoptimumkan peratusan berat MWCNT, masa sonikasi oleh komposit telah dioptimumkan. Ini dilakukan dengan menyediakan sampel 0.4wt % MWCNT yang kemudiannya telah disonikasi pada jangka masa yang berbeza. Selepas sonikasi, kelikatan ditentukan. Dari keputusan itu, kelikatan adalah yang tertinggi bagi sampel yang disonikasi untuk 90 minit. Oleh itu, masa sonikasi optimum untuk 0.4wt % MWCNT adalah 90 minit. Selepas pengoptimuman, komposit itu kemudian dianalisis dengan menggunakan ujian Kekuatan Tegangan (TS), terma Analisis Gravimetrik (TGA) dan Spektroskopi Inframerah Transformasi Fourier(FTIR). Daripada ujian Kekuatan Tegangan, ia boleh disimpulkan bahawa komposit dengan 0.4wt % MWCNT lebih kuat daripada sampel tanpa MWCNT. Hal ini menunjukkan bahawa komposit Poliester ini mempunyai potensi untuk digunakan dalam pelbagai aplikasi.

Kata Kunci: Poliester; Tiub Nano Karbon; Kelikatan; Serakan; Ultrasonikasi

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LIST OF ABBREVIATIONS

MWCNTs	Multi-Walled Carbon Nanotubes
MWCNT	Multi-Walled Carbon Nanotube
DWCNT	Double-Walled Carbon Nanotube
SWCNT	Single-Walled Carbon Nanotube
CNTs	Carbon Nanotubes
CNT	Carbon Nanotube
CB	Carbon Black
THF	Tetrahydrofuran
MEKP	Methyl Ethyl Ketone Peroxide
TGA	Thermal Gravimetric Analysis
FTIR	Fourier Transform Infrared Spectroscopy
TS	Tensile Strength
FKKSA	Fakulti Kejuruteraan Kimia & Sumber Asli
UMP	Universiti Malaysia Pahang

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

In recent years, much effort has been placed on producing materials with unusual combination of properties that cannot be met with conventional ceramics, metal alloys and polymeric materials. This material which is known as composite materials or composites are materials made from two or more constituent materials with relatively different properties, in which when combined, produces a new material with significantly improved properties that is different from the individual materials.

Composites are preferable due to its improved mechanical, physical and chemical properties. In addition, they are also stronger, lighter and might also be less expensive when compared to conventional materials such as ceramics, metal alloys and polymeric materials.

Composite materials are generally made up of two phases which are the matrix phase and the reinforcement phase. The individual components in the composite normally remain separate and distinct within the structure. Generally, the matrix phase is comprised of resins such as polyester or epoxy which are continuous phase whereas the reinforcement phase is made of various kinds of fibers. In this study, the resins used are Polyester Resin and the fiber used is Carbon Nanotube (CNT) which is Carboxyl (COOH) functionalized.

1.2 Motivation

Carbon Nanotube (CNT) has the potential to be accepted as advanced materials for the upcoming generation. This is due to its excellent properties such as high thermal conductivity, high mechanical strength and good electrical properties. Fundamental research progressed to date suggests that CNT is regarded as promising reinforcements in the polymer composites due to the combination of their uniquely excellent properties with high aspect ratio and small size (Ebbesen, 1994).

The discovery of multi-walled carbon nanotubes (MWCNTs) in 1991 has stimulated ever-broader research activities in science and engineering devoted entirely to carbon nanostructures and their applications (Iijima, 1991). Since the discovery of CNT in 1991 by Iijima, CNT has attracted a lot of researches to do research for its potential applications in a broad range of industry. For an instance, Ma et al. (2003) synthesized CNT/polymer systems using polyester. However, due to limited availability and higher cost, there are only a few practical applications in industrial field.

Dispersion of nanofillers plays a very important role in the use of filler properties in polymeric composites. Nanoparticles due to large surface area and mostly high aspect ratio tend to agglomerate greatly which reduces the ability to show their expected properties (Peigney et.al, 2001). A technique to achieve good dispersion of nanoparticles is ultra-sonication which can be used also for CNTs. Applying this method in a low-price polymer-like polyester which has good properties such as versatileness, quick curing, and low viscosity leads to fabrication of CNT/polyester composite with enhanced properties.

According to Aurilia et al. (2012), CNT addition into polymeric materials is a valuable solution in performances enhancement, when adequately dispersed. Nevertheless, in this research, the incorporation of CNT in polyester was studied due to the interest in improving the properties of the polyester for various applications.

1.3 Problem Statement

According to the literature, there were some drawbacks that were faced by various researches on producing Carbon Nanotube (CNT) reinforced Polyester composite materials. Firstly, CNT tends to bundle together and to form some agglomeration due to intrinsic van der Waals attraction between the individual tubes (Dresselhaus et al., 2001).

Secondly, weak interfacial bonding between the nanotubes and the polymer matrix has limited the efficient load transfer to the polymer matrix, playing a limited reinforcement role in the polymer nanocomposites (Lourie et al., 1998).

Hence, in this study, both problems would be eliminated or reduced by optimizing the weight percentage of CNT in the composite and by optimizing the sonarification time. This could produce a new composite with improved properties.

1.4 Objectives

There are two main objectives in this study:

a) To obtain the best dispersion of Carbon Nanotube (CNT) in Polyester resin
b) To produce Carbon Nanotube (CNT) Reinforced Polyester

1.5 Scope

This study will be focusing on the production of Carbon Nanotube (CNT) Reinforced Polyester and the characterization of the composite. The area of study narrows to the following:

a) To determine the optimum weight percentage of Carbon Nanotube (CNT) in the composite without using any solvent
b) To determine the optimum weight percentage of Carbon Nanotube (CNT) in the composite by using Tetrahydrofuran (THF) as solvent
c) To determine the optimum sonication time needed for Carbon Nanotube (CNT) & Polyester composite
d) To characterize the composite using various analysis such as Viscometer, FT-IR spectral analysis, Thermal Gravimetric Analysis (TGA), and Tensile Test.

CHAPTER 2

LITERATURE REVIEW

2.1 Composites

Many of our modern technologies require materials with unusual combinations of properties that cannot be met by the conventional metal alloys, ceramics, and polymeric materials. Material property combinations and ranges have been, and are yet being, extended by the development of composite materials. Generally speaking, a composite is considered to be any multiphase material that exhibits a significant proportion of the properties of both constituent phases such as that a better combination of properties is realized. (Callister & Rethwisch, 2008).

A composite, in the present context, is a multiphase material that is artificially made, as opposed to one that occurs or forms naturally. In addition, the constituent phases must be chemically dissimilar and separated by a distinct interface. Thus, most metallic alloys and many ceramics do not fit this definition because their multiple phases are formed as a consequence of natural phenomena.

In designing composite materials, scientists and engineers have ingeniously combined various metals, ceramics, and polymers to produce a new generation of extraordinary materials. Most composites have been created to improve combinations of mechanical characteristics such as stiffness, toughness, and high temperature strength.

Many composite materials are composed of just two phases; one is termed the matrix, which is continous and surrounds the other phase, often called the dispersed phase. The properties of composites are a function of the properties of the constituent phases, their relative amounts, and the geometry of the dispersed phase. “Dispersed phase geometry” in this context means the shape of the particles and the particle size, distribution, and orientation.

2.1.1 Fiber-Reinforced Composites

Technologically, the most important composites are those in which the dispersed phase is in the form of a fiber. Design goals of fiber-reinforced composites often include high strength or stiffness on a weight basis. These characteristics are expressed in terms of specific strength and specific modulus parameters, which correspond, respectively, to the ratios of tensile strength to specific gravity and modulus of elasticity to specific gravity. Fiber-reinforced composites with exceptionally high specific strengths and moduli have been produced that utilize low-density fiber and matrix materials. (Callister & Rethwisch, 2008).

2.1.1.1 Influence of Fiber Length

The mechanical characteristics of a fiber-reinforced composite depend not only on the properties of the fiber, but also on the degree to which an applied load is transmitted to the fibers by the matrix phase.

Some critical fiber length is necessary for effective strengthening and stiffening of the composite material. This critical length, l_c is dependent on the fiber diameter, d and its ultimate strength σ_f , and on the fiber-matrix bond strength (or the shear yield strength of the matrix, whichever is smaller) τ_c according to

$$l_c = \frac{\sigma f d}{2\tau c}$$

For a number of glass and carbon fiber-matrix combinations, this critical length is on the order of 1mm, which ranges between 20 and 150 times the fiber diameter. As fiber length l increases, the fiber reinforcement becomes more effective.

Fibers for which $l \gg l_c$ (normally $l > 15l_c$) are termed continous; discontinuous or short fibers have lengths shorter than this. For discontinuous fibers of lengths significantly less than l_c , the matrix deforms around the fiber in such a way that there is virtually no stress transference and little reinforcement by the fiber. For a significant improvement in strength of the composite, the fibers must be continuous.

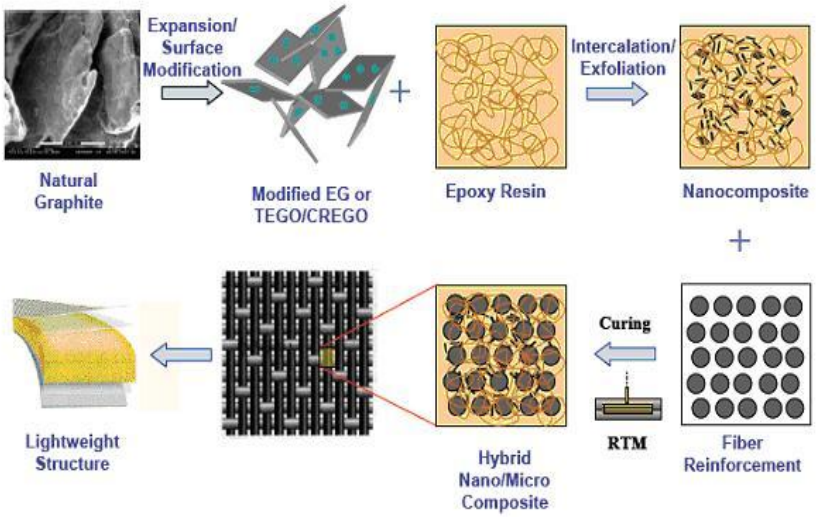


Figure 2.1 Fiber reinforcement in Epoxy Resin

2.1.1.2 Influence of Fiber Orientation

The arrangement or orientation of the fibers relative to one another, the fiber concentration, and the distribution all have significant influence on the strength and other properties of fiber-reinforced composites. With respect to orientation, two extremes are possible:

i) Continuous and Aligned Fiber Composites
ii) Discontinuous and Aligned Fiber Composites
iii) Discontinuous and Random Fiber Composites

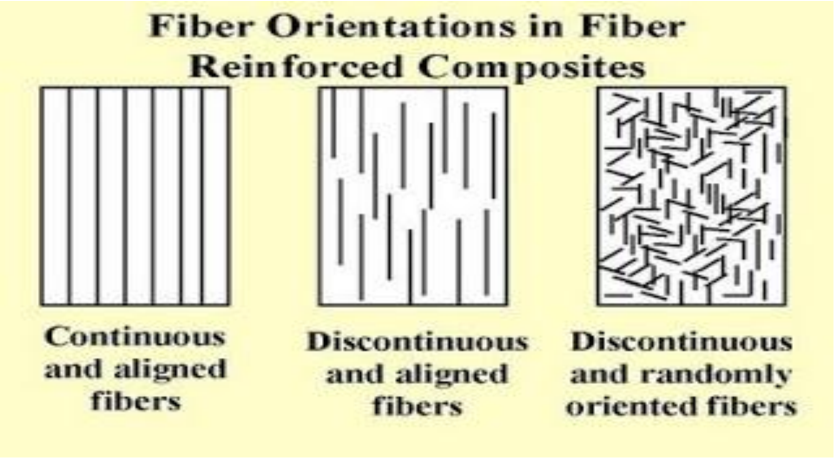


Figure 2.2 Fiber Orientations in Fiber-reinforced composites

2.2 Carbon Fiber-Reinforced Polymer (CFRP) Composites

Carbon is a high-performance fiber material that is the most commonly used reinforcement in advanced polymer-matrix composites. The reasons for this are as follows:

- Carbon fibers have the highest specific modulus and specific strength of all reinforcing fiber materials.
- They retain their high tensile modulus and high strength at elevated temperatures; high temperature oxidation, however, may be a problem.
- At room temperature, carbon fibers are not affected by moisture or a wide variety of solvents, acids, and bases.
- These fibers exhibit a diversity of physical and mechanical characteristics, allowing composites incorporating these fibers to have specific engineered properties.
- Fiber and composite manufacturing processes have been developed that are relatively inexpensive and cost effective.

Carbon fibers are not totally crystalline, but are composed of both graphitic and noncrystalline regions. Besides that, carbon fibers are normally coated with a protective epoxy size that also improves adhesion with the polymer matrix.

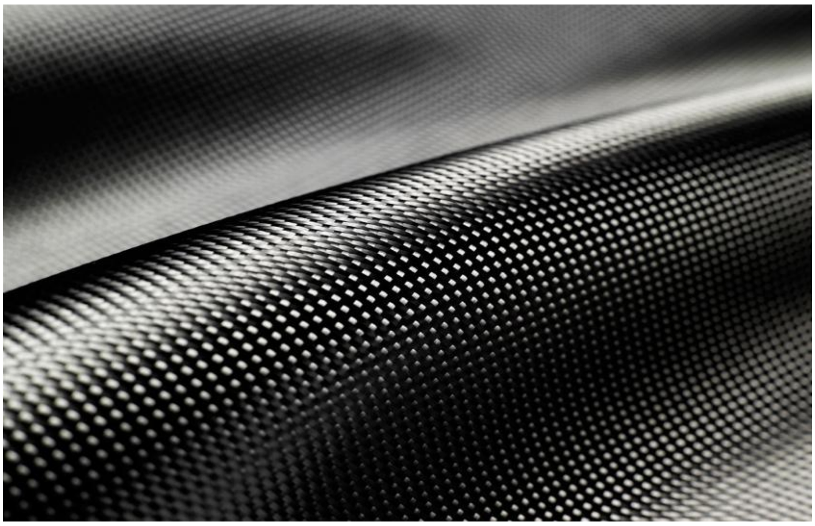


Figure 2.3 Application of Carbon Fiber in Automobile Industry

Carbon-reinforced polymer composites are currently being utilized extensively in sports and recreational equipments (fishing rods, golf clubs), filament-wound rocket motor cases, pressure vessels, and aircraft structural components- both military and commercial, fixed wing and helicopters (e.g., as wing, body, stabilizer, and rudder components). (Callister & Rethwisch, 2008).

2.3 Polyester Resin

Polyester resins are unsaturated resins formed by the reaction of dibasic organic acids and polyhydric alcohols. Polyester resins are used in sheet moulding compound, bulk moulding compound and the toner of laser printers. Wall panels fabricated from polyester resins reinforced with fiberglass — so-called fiberglass reinforced plastic (FRP) — are typically used in restaurants, kitchens, restrooms and other areas that require washable low-maintenance walls.

Unsaturated polyesters are condensation polymers formed by the reaction of polyols (also known as polyhydric alcohols), organic compounds with multiple alcohol or hydroxy functional groups, with saturated or unsaturated dibasic acids. Typical polyols used are glycols such as ethylene glycol; acids used are phthalic acid and maleic acid. Water, a by-product of esterification reactions, is continuously removed, driving the reaction to completion.

Unsaturated polyester resins are further classified into the following categories:

- Ortho-phthalic polyesters – resins made from ortho-phthalic anhydride are generally cheaper than the other two classes of unsaturated polyester resins. They are usually used to manufacture general purpose composite laminates where only moderate structural properties are required.
- Iso-phthalic polyesters – resins made from Iso-phthalic acid. These resins are much more structurally competent than the ortho-phthalic resins. They also have superior corrosion resistance and are used for more demanding applications
- Tere-phthalic polyesters – Tere-phthalate resins are made from tere-phthalic acid. These resins are currently made in small volumes and are considered a specialty resin. Although they tend to have better thermal and chemical resistance than iso-phthalic resins they are difficult to manufacture.

Figure 2.4 Polyester Resin

The use of unsaturated polyesters and additives such as styrene lowers the viscosity of the resin. The initially liquid resin is converted to a solid by cross-linking chains. This is done by creating free radicals at unsaturated bonds, which propagate in a chain reaction to other unsaturated bonds in adjacent molecules, linking them in the process. The initial free radicals are induced by adding a compound that easily decomposes into free radicals. This compound is usually and incorrectly known as the catalyst. Substances used are generally organic peroxides such as benzoyl peroxide or methyl ethyl ketone peroxide.

Recently there has been a global focus on greener processes and technologies. This is where unsaturated polyesters have the most potential over their metallic counterparts. Production of metals continues to deplete our natural resources and consumes most of our fossil fuels. Unsaturated polyesters although previously synthesized from fossil fuels can now be manufactured from biological resources instead. Starches, plant oils and other naturally derived building blocks have been discovered that can be used to prepare unsaturated polyester resins.

2.3.1 Characteristics of Polyester Resin

The material has the potential to be 100 percent solid. This depends on how fast the reaction takes place. The styrene is volatile prior to the reaction. Heat is not typically added to the system except when cure time is expected to be long, such as on cool spring or fall days. The catalyst is added to drive the reaction. Usually, the catalyst is methyl ethyl ketone (MEK) or benzoyl peroxide. The polyester resin and the styrene solvent react together to crosslink, or polymerize, to form a film. The polyester resin system will not cure properly if the appropriate quantity of catalyst is not added.

Unsaturated polyester resins also have excellent service temperatures. They have good freeze-thaw resistance and can be designed for use in many low to moderate temperature applications ranging from refrigerated enclosures to hot water geysers.

2.3.1.1 Advantages of Polyester Resin

Polyester resin is the resin that mainly preferred in industries; this is because it got a lot advantages. The advantages are:

Advantages

- Essentially two components in one container
- Long lasting and durable
- Does not discolor badly
- Relatively inexpensive
- Works well on concrete

2.3.1.2 Disadvantages of Polyester Resin

Although, there are a lot advantages of using polyester resin, it still has some disadvantages. The disadvantages are:

Disadvantages

- Peroxide catalyst is a very reactive oxidizer
- Requires placarding as a hazardous material

- Requires commercial drivers license
- Flush solvent is flammable and a hazardous waste
- Moisture in surface a major factor and detriment
- Set up time depends on type of resin (usually 3-20 minutes)
- Difficult to determine whether mixed properly

2.3.2 Classification of Polyester

Polyesters were broadly classified into unsaturated and saturated polymers. These were two broad divisions subdivided as follows: (Bruins, 1976)

1. Unsaturated

a) Laminating and Casting Resins. These were based on dibasic acids and dihydric alcohols. The polyester unit formed must be capable of copolymerizing with a vinyl-type monomer, thereby yielding a vinyl-polyester copolymer or simply cured polyester having a thermoset structure.

b) Alkyds. In general, the same types as (a) although the glyptal (surface coatings) types are modified with oils or fatty acids. This term was also used to describe a group of thermosetting moulding materials based on the reaction of a dihydric alcohol with an unsaturated acid such as maleic in place of the conventional phthalic acid. A vinyl type monomer was also necessary to affect speedy cross-linking and curing and used as moulding powders for compression and transfer-moulding techniques.

2. Saturated

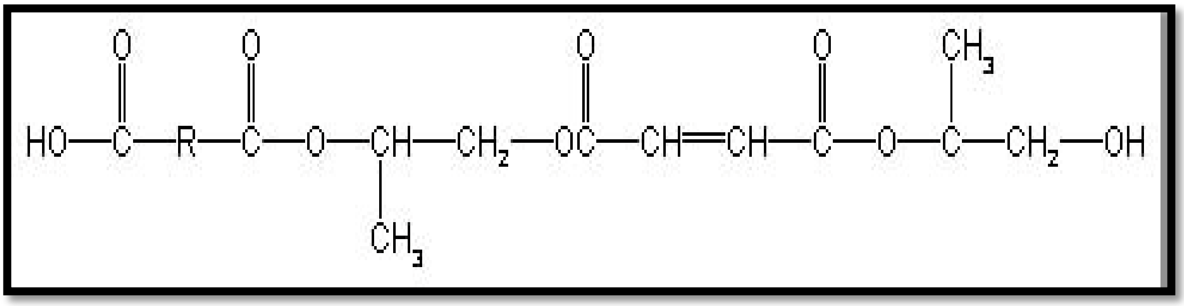
a) Fibres and Films. These were based on the reaction of terephthalic acid with ethylene glycol and were linear, high molecular weight polymers which do not undergo any cross-linking reactions.

b) Plasticizers. These were polyesters which were completely saturated, normally referred to as polymeric plasticizers.

c) Polyester/ Polyurethanes. Certain polyesters having high hydroxyl content were reacted with various isocyanates to form polyurethanes, which were finding extensive use as foams, elastomers, surface coatings and adhesives.

2.3.3 Synthesis of Polyester Resin

Polyesters were polymers with repeating carboxylate groups in their backbone chain. The polyester backbone was generally composed of three basic types of structural units, saturated acids, unsaturated acids and glycols (Figure 2.5).



$$HO-\overset{\overset{O}{\|}}{C}-R-\overset{\overset{O}{\|}}{C}-O-\underset{\underset{CH_3}{|}}{CH}-CH_2-O\overset{\overset{O}{\|}}{C}-CH{=}CH-\overset{\overset{O}{\|}}{C}-O-\overset{\overset{CH_3}{|}}{C}-CH_2-OH$$

Figure 2.5: Anhydrides Acid, Acids, Unsaturated Anhydrides Acids and Alcohols Formed Polyester. Birkley, (1989)

In the case of the general-purpose polyester, these components usually consisted of phthalic acid, maleic acid, and propylene glycol. This was a very desirable composition since it had the characteristics of low cost and good quality; propylene glycol and phthalic anhydride were the lowest cost raw material in their class. The unsaturated acid provided the sites for cross-linking, the saturated acid determined the degree of spacing and the glycol, of course, provided the means for esterification and for bridging the acids to form a polymer. Linear unsaturated polyesters were prepared batch wise by heating and cooling and fitted for distillation. A typical formulation for a general-purpose material were as follows; propylene glycol, 100 parts by weight, maleic anhydride, 72 parts by weight and phthalic anhydride 54 parts by weight.

First produced commercially in the late 1920s, polyesters have become important compounds used in a wide variety of industries. The most economically important types of polyesters include poly (ethylene terephthalate) (PET) and poly (butylene terephthalate) (PBT). The chemical reactions for making polyesters were investigated in 1901 and resulted in the production of glyptal polyesters. These reactions involved the combination of a diacid with a diol. The reaction was called a condensation reaction because the two initial types of monomers combined to produce a longer chain polymer and water as a by product.

Linear polyesters were not produced until the 1930s, when W. H. Carothers systemically investigated reactions of diols with diacids. Carothers was not successful in producing a polyester fiber and switched the focus of his research. In 1942, John Whinfield and W. Dickson made the first high molecular weight PET. After these fibers were produced other polyesters were discovered and have since become very important compounds (Beier et.al, 2007).

Linear unsaturated polyesters were prepared commercially by the reaction of a saturated diol with a mixture of an unsaturated dibasic acid and a modifying dibasic acid. In principle, unsaturation desired in a polyester can be derived from either an unsaturated diol or an unsaturated acid provides sites for subsequent cross-linking; the function of the modifying acid was to reduce the number of reactive unsaturated sites along the polymer and hence to reduce the cross-link intensity and brittleness of the final product. Some acids and anhydrides which were used to modify polyester are, in fact, unsaturated but the double bonds were not sufficiently reactive to represent sites for subsequent cross-linking (Saunders, 1988).

Unsaturated polyester resins were addition products of various saturated acids, unsaturated acids and glycols. Many patents for the preparation of these products were issued within the past 30 years. The polymers making up this first group of polyesters were linear polyesters containing aliphatic unsaturation which provided sites for subsequent cross-linking. A polymer of this type first became available in the U.S.A in 1946; the polymer was prepared from diethylene glycol and maleic anhydride and could be crosslinked by reaction with styrene (Saunders, 1999).

2.3.4 Current Interest Research of Unsaturated Polyester

Polyesters were important class of high performance and engineering polymers, which find use in a number of diverse applications (Barbiroli et al., 2003; Aziz et al., 2005; Qazvini & Mohammadi, 2005). Unsaturated polyester resin were chosen first for making fibre reinforced plastics (FRP) by any molder because of the ease of handling and fabrication and the low cost as compared to epoxy resin. They were primarily used in compression molding (sheet molding compounds), injection molding (bulk molding compounds), resin transfer molding, pultrusion, filament winding and hand lay-up process (Vilas et al., 2001). About 85% of the FRP products like boats, car and aircraft components and chairs) were manufactures using polyesters (Devi et al., 1997).

Different kinds of polyesters were synthesized over the past decades from various types of diacid chlorides and diols. Thermally stable polyesters derived from isophtalic and terephthalic acids with bisphenol-A was commercialized (Ramakrishna, 2005). However, polyesters were generally difficult to process because of their limited solubility in organic solvents and their melting temperature or high glass-transition temperature by virtue of their rigid structures (Tamami et al., 2004). Therefore development of polyesters for use at high temperature with improved solubility was an important goal.

One of the approaches to improved solubility as well as processability of the polymers without extremes loss of their high thermal stability was the introduction of polar and flexible groups into the polymer backbone (Messori et al., 2001; Ito et al., 2001; He et al., 2001; Aziz et.al, 2004; Huang & Jiang, 1998). The incorporation of bulky pendent groups were also provided beneficial effect for solubility because this approach produces a separation of chains and lowering the chain packing with a molecular mobility, so that the overall observable effect improvement of solubility at the same time (Tsubokawa et al., 1983; Xueqiu et al., 1988; Walter et al., 1995; Lange et al., 1997; Grobelny, 1997). It was well known that a large number of polymers containing heterocyclic ring in the main chain were resistant to high temperature (Blumstein et al., 1982).

Recently, the researchers synthesized new polyesters containing rigid segment such as pyridine rings that possess high glass transition temperatures and enhanced solubility in organic solvents (Chen et al., 2005). Current interest in unsaturated polyester (UP) resins also focused on the enhancement of chemical inertness, solvent and high temperature resistant, barrier properties, low friction coefficient and low surface tension, that in principle were transferred to other polymeric materials by blending or copolymerization. Some enhancement such as excellent resistance to corrosion, water and atmospheric agents, formulations for resins and foams and several others were also reported in patent literature (Messori et al., 2001).

Unsaturated copolyesters, also called polyester resins, were based on macromolecules with a polyester backbone in which both a saturated acid and unsaturated acid were condensed with a dihydric alcohol (Qazvini & Mohammadi, 2005). A survey of the scientific literature reveals that few unsaturated copolyesters based on the interaction of unsaturated diols and saturated acids were synthesized and studied (Tiitu et al., 2005). Imai and Tassavori synthesized and studied aromatic polyesters and copolyesters containing phenylindane units with Tg 235- 253ºC (Messori et al., 2001).

The present investigation deals with the synthesis and characterization of new unsaturated polyesters and co-polyesters based on some dibenzylidenecycloalkanones and containing meta- and para-azo groups in the main chain. A major purpose of this work was to investigate the effect of a cycloalkanone ring, in the polymer backbone, on polymer properties.

2.4 Carbon Nanotube (CNT)

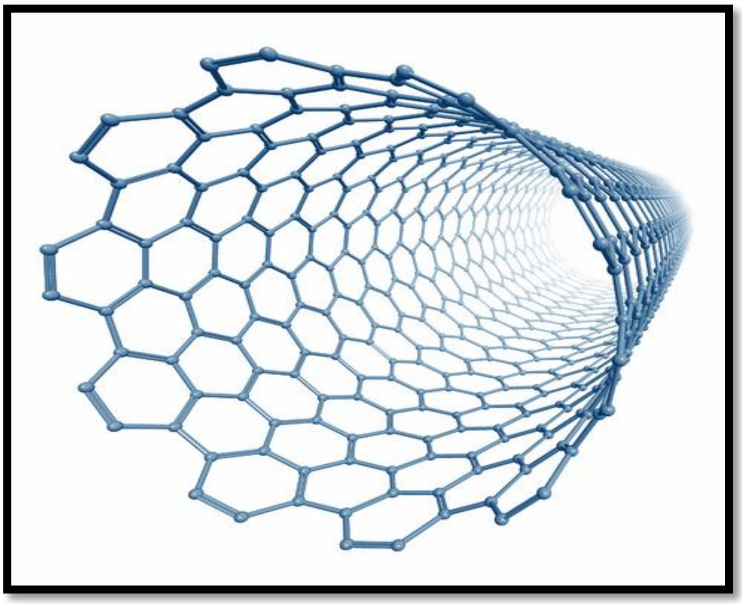
In 1981 a group of Soviet scientists published the results of chemical and structural characterization of carbon nanoparticles produced by a thermocatalytical disproportionation of carbon monoxide. Using TEM images and XRD patterns, the authors suggested that their “Carbon multi-layer tubular crystals” were formed by rolling graphene layers into cylinders.

The last few years have witnessed the discovery, development and, in some cases, large-scale manufacturing and production of novel materials that lie within the nanometer scale. Such novel nanomaterials consist of inorganic or organic matter and in most cases have never been studied in the context of pharmaceuticals. Carbon verynanotubes (CNTs) are one of them. CNTs are allotropes of carbon. They are tubular in shape, made of graphite. CNTs possess various novel properties that make them useful in the field of nanotechnology and pharmaceuticals. They are nanometers in diameter and several millimeters in length and have a very broad range of electronic, thermal, and structural properties. These properties vary with kind of nanotubes defined by its diameter, length, chirality or twist and wall nature. Their unique surface area, stiffness, strength and resilience have led to much excitement in the field of pharmacy (Lacerda et.al, 2006).

Dispersion of nanofillers plays a very important role in the use of filler properties in polymeric composites. Nanoparticles due to large surface area and mostly high aspect ratio tend to agglomerate greatly which reduces the ability to show their expected properties (Peigney et.al, 2001). A technique to achieve good dispersion of nanoparticles is ultra-sonication which can be used also for CNTs. Applying this method in a low-price polymer-like polyester which has good properties such as versatileness, quick curing, and low viscosity leads to fabrication of CNT/polyester composite with enhanced properties.

2.4.1 Single walled Carbon Nanotube (SWCNT)

Single-walled carbon nanotubes (SWNTs) are nanometer-diameter cylinders consisting of a single graphene sheet wrapped up to form a tube. Since their discovery in the early 1990s (Iijima & Ichihasi, 1993) (Bethune et.al, 1993), there has been intense activity exploring the electrical properties of these systems and their potential applications in electronics. Experiments and theory have shown that these tubes can be either metals or semiconductors, and their electrical properties can rival, or even exceed, the best metals or semiconductors known. Particularly illuminating have been electrical studies of individual nanotubes and nanotube ropes (small bundles of individual nantoubes). The first studies on metallic tubes were done in 1997 (Tans et.al, 1997) (Cobden et.al, 1998) and the first on semiconducting tubes in 1998 (Tans et.al, 1998).



.**Figure 2.6 Single Walled Carbon Nanotube (SWCNT)**

2.4.2 Multiwalled Carbon Nanotube (MWCNT)

The discovery of multi-walled carbon nanotubes (MWCNTs) in 1991 has stimulated ever-broader research activities in science and engineering devoted entirely to carbon nanostructures and their applications (Iijima, 1991). This is due in large part to the combination of their expected structural perfection, small size, low density, high stiffness, high strength (the tensile strength of the outer most shell of MWCNT is approximately 100 times greater than that of aluminium), and excellent electronic properties. As a result, carbon nanotubes (CNT) may find use in a wide range of applications in material reinforcement, field emission panel display, chemical sensing, drug delivery, and nanoelectronics.

Multiwalled carbon nanotubes consist of several seamlessly rolled up graphene sheets, with typical shell or wall separations, dwall, of 0.34 nm (Duesberg et al. 1998). In the absence of mechanical entanglements, MWNTs tend to exist as individual particles in suspensions with many organic liquids. Carbon nanotubes are extremely strong, 30-100 times stronger than steel, but with only one sixth of its weight.

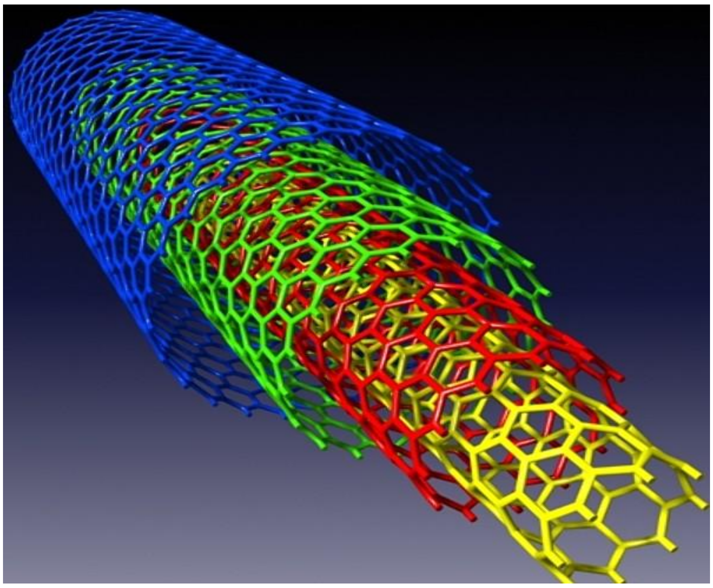


Figure 2.7 Multi walled Carbon Nanotube (MWCNT)

2.4.3 Comparison between SWCNT and MWCNT

The comparison between Single walled Carbon nanotube and Multi walled Carbon Nanotube had been done by Rajashree Hirlekar et.al in 2009. The details are provided in Table 1.

Table 2.1: Comparison between SWCNT and MWCNT

No.	SWCNT	MWCNT
1	Single layer of graphene	Multiple layer of grapheme
2	Catalyst is required for synthesis	Can be produced without catalyst
3	Bulk synthesis is difficult as it requires proper control over growth and atmospheric condition	Bulk synthesis is easy
4	Purity is poor	Purity is high
5	A chance of defect is more during functionalization	A chance of defect is less but once occurred it’s difficult to improve
6	Less accumulation in body	More accumulation in body
7	Characterization and evaluation is easy	It has very complex structure
8	It can be easily twisted and more pliable	It cannot be easily twisted

2.4.4 Properties of Carbon Nanotube (CNT)

Carbon nanotube is one of the strongest materials in nature. Carbon nanotubes (CNTs) are basically long hollow cylinders of graphite sheets. Although a graphite sheet has a 2D symmetry, carbon nanotubes by geometry have different properties in axial and radial directions. It has been shown that CNTs are very strong in the axial direction (Yu et.al, 2000). Young's modulus on the order of 270 - 950 GPa and tensile strength of 10 - 60 GPa were obtained.

The intrinsic mechanical and transport properties of Carbon Nanotubes make them the ultimate carbon fibers. The following tables (Table 2 and Table 3) compare these properties to other engineering materials.

Overall, Carbon Nanotubes show a unique combination of stiffness, strength, and tenacity compared to other fiber materials which usually lack one or more of these properties. Thermal and electrical conductivity are also very high, and comparable to other conductive materials.

Table 2.2: Mechanical Properties of Engineering Fibers

Fiber Material	Specific Density	E (TPa)	Strength (GPa)	Strain at Break (%)
Carbon Nanotube	1.3 - 2	1	10 - 60	10
HS Steel	7.8	0.2	4.1	< 10
Carbon Fiber - PAN	1.7 - 2	0.2 - 0.6	1.7 - 5	0.3 - 2.4
Carbon Fiber - Pitch	2 - 2.2	0.4 - 0.96	2.2 - 3.3	0.27 - 0.6
E/S – glass	2.5	0.07 / 0.08	2.4 / 4.5	4.8
Kevlar* 49	1.4	0.13	3.6 - 4.1	2.8

Kevlar is a registered trademark of DuPont.

Table 2.3: Transport properties of Conductive Materials.

Material	Thermal Conductivity (W/m.k)	Electrical Conductivity
Carbon Nanotubes	> 3000	106 – 107
Copper	400	6 x 107
Carbon Fiber - Pitch	1000	2 - 8.5 x 106
Carbon Fiber - PAN	8 – 105	6.5 - 14 x 106

2.4.5 Dispersion of CNTs

The objective of dispersion science and technology is to produce a suspension of independent, separated nanotubes that then can be manipulated into preferred orientations in one-dimensional (fiber), two dimensional (flat sheet), or three-dimensional (bulk solid) objects. There are two different approaches to nanotube dispersion:

a) Mechanical (or physical) methods
b) Chemical methods.

2.4.5.1 Mechanical (or physical) methods

Mechanical dispersion methods, such as ultrasonication, separate nanotubes from each other, but can also fragment the nanotubes, decreasing their aspect ratio during processing.

2.4.5.2 Chemical methods

Chemical methods use surfactants or functionalization to change the surface energy of the nanotubes, improving their wetting or adhesion characteristics and reducing their tendency to agglomerate in the continuous phase solvent. However, aggressive chemical functionalization, such as using neat acids at high temperatures, can digest the nanotubes. Both mechanical and chemical methods can alter the aspect ratio distribution of the nanotubes, resulting in changes in the properties of their dispersions.

2.4.5.3 Ultrasonication of CNTs

Ultrasonication of carbon nanotubes in solvents such as alcohols is a common technique for dispersing samples for electron microscopy. One way to improve the dispersion of nanotubes is to shorten the tubes. The shorter tubes are less likely to entangle and arrange into aggregates. However, there are some serious disadvantages with breaking the tubes into smaller pieces. When the tube-walls are broken in order to create a cut, the wall may become damaged in other ways as well.

There are two major methods for delivering ultrasonic energy into liquids, the ultrasonic bath and the ultrasonic horn or wand. Ultrasonication disperses solids primarily through a bubble nucleation and collapse sequence.

The ultrasonication bath has a higher frequency (40–50 kHz) than cell dismembrator horns (25 kHz). Ultrasonication of fluids leads to three physical mechanisms: cavitation of the fluid, localized heating and the formation of free radicals. Cavitation, the formation and implosion of bubbles, can cause dispersion and fracture of solids. The frequency of the ultrasound determines the maximum bubble size in the fluid. Low frequencies (about 20 kHz) produce large bubbles and high energy forces occur as they collapse. Increasing the frequency reduces bubble size and nucleation, so that cavitation is reduced. Cavitation does not occur in many liquids at frequencies larger than 2.5 MHz. The ultrasonication bath does not produce a defined cavitation zone as does a horn and the energy seems to be more uniformly dispersed through the liquid phase. Systems with low frequencies (20–100 kHz) and high power (100–5000 W) are used to modify materials.

Figure 2.8 Ultrasonicator used for ultrasonication bath

2.4.6 Current/Short-term applications of MWCNT

Current or short-term applications are often based on the use of MWCNTs as a superior replacement of electrically conductive carbon blacks. Much of the history of plastics over the last half century has been a replacement for metal. For structural applications, plastics have made tremendous headway. However where electrical conductivity is required, metals are still preferred to plastics. This deficiency can be overcome by upgrading plastics with conductive fillers such as carbon black and graphite fibres. However the loading required to provide the necessary conductivity is typically high, and the structural properties of the resulting plastic parts are highly degraded. Due to their high conductivity, high aspect ratio, and natural tendency to form ropes, MWCNTs are ideal in providing inherently long conductive pathways even at ultra-low loadings. The lower loading of additive can offer several advantages such as better processability or higher retention of the mechanical properties of the original polymer. This is why the use of carbon nanotubes for antistatic and conductive applications in polymers is already a commercial reality, growing in sectors such as electronics and the automotive industry. For these applications, carbon nanotubes can compete with additives such as highly conductive carbon black on a price performance basis.

Applications that exploit this behaviour of CNTs include EMI/RFI (electromagnetic and radio frequency interference) shielding composites, electrostatic dissipation (ESD), antistatic materials and (even transparent!) coatings. Concrete examples in the automotive industry are fuel systems components and fuel lines (connectors, pump parts, o-rings, …), exterior body parts for electrostatic painting as well as, in the electronic industry, conveyor belts, manufacturing tools and equipments, wafer carriers, clean room equipments, etc.

Structural composites made of carbon fiber (or glass fiber) and a thermoset (e.g. epoxide) have been improved quite substantially by the introduction of carbon nanotubes. The benefit is not achieved by replacing the reinforcing carbon fiber but by enhancing the properties of the matrix material (epoxide). Almost every sports item on the market can be improved by using CNT. These high-end models are usually used by professional athletes as they are often lighter weighted and more durable. For examples, the tennis champion Roger Federer is playing with CNT reinforced rackets while the famous national Finnish ice hockey team is equipped with CNT reinforced sticks.

These new structural composite materials based on CNT reinforced thermoplastics or thermosets combine low density and strong mechanical properties and will open the way to new developments in particular by replacing metals in various mechanical applications where a weight reduction could save energy, like in automotive industry. Moreover thanks to the good thermal conductivity of CNT, it is possible to develop composite materials able to quickly dissipate the heat and consequently to act as protective equipment in various fields.

2.4.7 Medium to Long term applications of MWCNT

Several running applications will be expanded to other industries. For example, the improvement of mechanical properties in epoxy-glassfiber or epoxy–carbon fiber composites already known from the sport industry can also be used in the construction of light weighted composites for wind power generators and in the aircraft industry. Due to the nature of these industries, more technical testing and longer certification time will be required. Other medium term applications may include electrical conductive inks for printable circuits, low cost RFID tags or antennas in cars. In the longer term, Carbon Nanotubes may also play a role in the modification of existing textile materials using electrostatic self-assembly and atomic layer deposition techniques to create novel and customizable surfaces on conventional textile materials with emphasis on natural fibres. This opens the way to the development of smart and intelligent textiles that combine new innovative functions.

Current/Short term	**Mid-term**	**Long term**
Conductive polymers & composites (automobiles and electronics)	Coatings (conductive thin films)	Microwave antennas
Sensors and Instruments (microscope probe tips, gas leak detectors)	Catalysts (petrochemical)	Seld-assembling yarns
Electromagnetic shielding	Textiles & fibres	Aerospace
Sporting goods (tennis rackets)	Lithium ion batteries	Medical implants
	Membrane and fibres	
	Lamps	
	Semiconducting materials	
	Advanced ceramics	
	Fuel Cells	
	Caulks and sealants	

Table 2.4: Key potential applications of MWCNTs on the short, mid and long term

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

In this chapter, the method of producing Carbon-nanotube (CNT) reinforced polyester will be discussed further. The CNT reinforced polyester will be characterized to determine its various properties. The characterization will be done by Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA), and Ultimate Tensile Testing Machine. In addition, other important information which will also be determined is the viscosity, sonification time, and drying time.

3.2 Glassware & Apparatus

2 beaker (50mL), 2 beaker (100mL), 2 beaker (250mL), 1 glass rod, 1 spatula, 10 vials, 1 measuring cylinder (250mL), 1 measuring cylinder (25ml), 1 measuring cylinder (10mL), 5 sample bottles, 6 petri dish (glass), 6 magnetic stirrer, 1 filter funnel, 5 seal bags, 1 vials rack, and 1 thermometer.

3.3 Material Description

5L polyester resin, 4L acetone with 99 % purity, 2L tetrahydrofuran with 99 % purity, 500 mL methyl ethyl ketone peroxide with 99 % purity and 500g Carbon nanotube (carboxyl group).

3.3.1 Polyester Resin

The primary material for this study is polyester resin. Polyester resin is a common chemical ordered from chemical companies. However, for this study, polyester resin is supplied by Universiti Malaysia Pahang (UMP).

Polyester resins are unsaturated resins formed by the reaction of dibasic organic acids and polyhydric alcohols. Polyester resins are used in sheet moulding compound, bulk moulding compound and the toner of laser printers. Wall panels fabricated from polyester resins reinforced with fiberglass — so-called fiberglass reinforced plastic (FRP) — are typically used in restaurants, kitchens, restrooms and other areas that require washable low-maintenance walls.

Unsaturated polyesters are condensation polymers formed by the reaction of polyols (also known as polyhydric alcohols), organic compounds with multiple alcohol or hydroxy functional groups, with saturated or unsaturated dibasic acids. Typical polyols used are glycols such as ethylene glycol; acids used are phthalic acid and maleic acid. Water, a by-product of esterification reactions, is continuously removed, driving the reaction to completion.

Figure 3.1 Polyester resin

3.3.1.1 Physical and Chemical Properties of Polyester resin

Appearance	A colourless viscous liquid with strong odour
Vapor Pressure	Less than 1.0
Boiling Point (°C)	135 - 140 °C
Density	1.15
Solubility in water	Immiscible
Flash Point (°C)	31 °C
Flammability limits	Lower Explosive limit = 1.0 %
Auto Ignition (°C)	480 °C
Volatile Components	Styrene

3.3.1.2 Handling and Storage of Polyester resin

Handling	Keep out of reach of children. Avoid unnecessary contact with the material. After use before eating, drinking or smoking, wash all exposed skin with soap and water.
Storage	Containers must be clearly labelled, rigid and strong. Store upright in a cool, dry, well ventilated area from heat, ignition sources and direct sunlight.

3.3.2 Acetone

Acetone is provided by Universiti Malaysia Pahang. A large amount of acetone is needed for this research because all the apparatus and glasswares used cannot be washed by water. The glasswares will become sticky because polyester resin does not dissolve in water. This will make the glassware cannot be used for further research. Thus, glasswares are cleaned and washed using acetone. Acetone should be handled with care, gloves and googles should be used to avoid any harm from acetone.

Figure 3.2 Acetone

3.3.2.1 Physical and Chemical properties of Acetone

Appearance	Liquid
Odor	Fruity. Mint-like. Ethereal
Taste	Pungent, Sweetish
Molecular Weight	58.08 g/mole
Colour	Colourless. Clear.
Boiling Point	56.2 °C
Melting Point	-95.35 °C
Critical Temperature	235 °C
Specific Gravity	0.79
Vapor Pressure	24 kPa
Odor threshold	62 ppm

3.3.2.2 Handling and Storage of Acetone

Precautions

Keep locked up. Keep away from heat. Keep away from source of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Wear suitable protective clothing. 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 oxidizing agents, reducing agents, acids, alkalis.

Storage

Store in a segregated and approved area (flammables area) . Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Keep away from direct sunlight and heat and avoid all possible sources of ignition (spark or flame).

3.3.3 Carbon Nanotube (CNT)

Carbon Nanotube (CNTs) is another primary material for this study. CNT exist as a black coloured powder. The CNT used for this study Carboxyl (COOH) functional group. Carbon Nanotubes were manufactured in China and were provided by Universiti Malaysia Pahang (UMP) for the purpose of this study.

Figure 3.3 Carbon Nanotube

3.3.3.1 Physical and Chemical Properties of Carbon Nanotube

Description	Carboxylic acid functionalized
Appearance	Dark granular solid
Odour	Odourless
Assay	> 80% carbon basis
Form	Powder
Avg.diam x Length	9.5 nm x 1.5µm

3.3.3.2 Handling and Storage of Carbon Nanotube

Handling

Do not breathe dust. Avoid contact with eyes, skin, and clothes. Avoid prolonged or repeated exposure.

Storage

Keep tightly closed and store in a cool and dry place.

3.3.4 Tetrahydrofuran (THF)

Tetrahydrofuran is the solvent used to disperse the carbon nanotubes in this research. It is proven that with addition of tetrahydrofuran enables the carbon nanotubes to disperse more in polyester resin. For this research, tetrahydrofuran is bought from Impian Z Enterprise.

Figure 3.4 Tetrahydrofuran

3.3.4.1 Physical and Chemical properties of Tetrahydrofuran

Physical state/ Appearance	Liquid
Odor	Ethereal. Fruity
Taste	Pungent
Molecular Weight	72.11 g/mole
Colour	Colourless
Boiling point	65 °C
Melting point	-108.3 °C
Critical Temperature	267 °C
Specific gravity	0.8892
Vapor Pressure	19.3 kPa
Vapor Density	2.5
Volatility	100 % (v/v)
Solubility	Soluble in diethyl ether, acetone. Partially soluble in cold water. Solubility in water is 30%.

3.3.4.2 Handling and Storage of Tetrahydrofuran

Precautions

Keep away from heat. Keep away from sources of ignition. Wear suitable protective clothing. 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 oxidizing agents.

Storage

Store tetrahydrofuran in a segregated and approved area. Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame). Prolonged exposure to air and light may form unstable explosive peroxides unless it is inhibited against peroxide formation.

3.3.5 Methy Ethyl Ketone Peroxide (MEKP)

Methyl ethyl ketone peroxide is the hardener used in this research. MEKP will be added to mixture of carbon nanotube and polyester resin. After stirring for 2 minutes, the solution then will be poured into metal mould to obtain “Dumbell shape” and “Bar shape”. MEKP will turn liquid composite to solid polymer by initiating cross-linking chain.

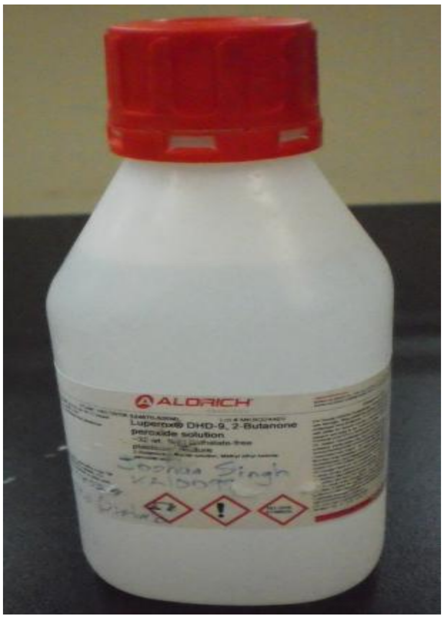


Figure 3.5 Methyl Ethyl Ketone Peroxide (MEKP)

3.3.5.1 Physical and Chemical properties of MEKP

Appearance	Clear water-white liquid
Odour	Slightly pungent odour
Melting point	Liquid at normal temperature
Flash point	82 °C
Specific gravity	1.11
Solubility in water	Less than 1% at 25 °C
Corrosiveness	Not corrosive

3.3.5.2 Handling and Storage of MEKP

Storage

Keep containers closed to prevent contamination. Rotate stock using the oldest material first. The activity and stability of MEKP is directly related to the shipping and storage temperature history. Cool storage at 80°F or below is recommended for longer shelf life and stability. Prolonged storage at elevated temperatures of 100°F and higher will cause product degradation, gassing and potential container rupture which can result in a fire and/or explosion.

Precautions

MEKP should never be added to hot solvents or monomers as a violent decomposition and/or reaction may result. When using spray equipment, never spray raw MEKP onto curing or into raw resin or flues. Keep MEKP in its original container.

3.4 Experiment

3.4.1 To determine the optimum weight percentage of Carbon Nanotube (CNT) in the composite

In this section, the optimum weight percentage of Carbon Nanotube (CNT) in the composite was determined without using any solvent.

1) Carbon Nanotube (CNT) powder was dried for one hour in an preheated oven at 105°C to remove any moisture content in it.
2) A sample with a total volume of 50mL was prepared. The sample was prepared by weight percentage of 0.1% CNT and 99.9% of Polyester Resin.
3) Both the measured CNT and Polyester Resin were added into a 100mL beaker and stirred using glass rod for about 1-2 minutes.
4) After that, the mixture was stirred using magnetic stirrer at full speed for about 10 minutes. No heating was required at this stage.
5) Then, the beaker was placed in an Ultrasound Water Bath for about 1 hour. No heating was required at this stage.
6) After 1 hour, the solution was tested to determine its viscosity using Viscometer.
7) After testing, the solution was kept in vials for further use.
8) The procedure from 2-7 was repeated for different percentage of CNT which was 0.2%, 0.3%, 0.4%, & 0.5%.
9) The results from Viscometer for each shear rate were recorded in the table below.

Trial	% CNT	CNT (g)	Torque (%)	Viscosity (mPa.s)	Str Rate (N/m^2)	Shear Rate (1/sec)
1	0.1	0.05				
2	0.2	0.10				
3	0.3	0.15				
4	0.4	0.20				
5	0.5	0.25				

3.4.2 To determine the optimum weight percentage of Carbon Nanotube (CNT) in the composite by using Tetrahydrofuran (THF) as solvent

In this section, the optimum weight percentage of Carbon Nanotube (CNT) in the composite was determined by using Tetrahydrofuran (THF) as solvent.

1) Carbon Nanotube (CNT) powder was dried for one hour in an preheated oven at 105°C to remove any moisture content in it.
2) A sample with a total volume of 50mL was prepared. The sample was prepared by weight percentage of 0.1% CNT and 99.9% of Polyester Resin.
3) Both the measured CNT and Polyester Resin were added into a 100mL beaker. In addition, Tetrahydrofuran (THF) which acts as a solvent was added into the beaker (Ratio of CNT to THF is 1:25).
4) The mixture was stirred using glass rod for 1-2 minutes.
5) After that, the mixture was stirred using magnetic stirrer at full speed for about 10 minutes. No heating was required at this stage.
6) Then, the beaker was placed in an Ultrasound Water Bath for about 1 hour. No heating was required at this stage.
7) After 1 hour, the solution was tested to determine its viscosity using Viscometer.
8) After testing, the solution was kept in vials for further use.
9) The procedure from 2-7 was repeated for different percentage of CNT which was 0.2%, 0.3%, 0.4%, & 05%.

10) The results from Viscometer for each shear rate were recorded in the table below.

Trial	% CNT	CNT (g)	Torque (%)	Viscosity (mPa.s)	Str Rate (N/m^2)	Shear Rate (1/sec)
1	0.1	0.05				
2	0.2	0.10				
3	0.3	0.15				
4	0.4	0.20				
5	0.5	0.25				

3.4.3 To determine the optimum sonication time needed for Carbon Nanotube (CNT) and Polyester composite

In this section, the optimum sonication time needed for Carbon Nanotube (CNT) & Polyester composite was studied.

1) Carbon Nanotube (CNT) powder was dried for one hour in an preheated oven at 105°C to remove any moisture content in it.
2) 5 samples with a total volume of 50mL each were prepared. The sample was prepared by weight percentage of 0.4% CNT and 99.6% of Polyester Resin.
3) Both the measured CNT and Polyester Resin were added into a 100mL beaker. In addition, Tetrahydrofuran (THF) which acts as a solvent was added into the beaker (Ratio of CNT to THF is 1:25).
4) The mixture was stirred using glass rod for 1-2 minutes.
5) After that, the mixture was stirred using magnetic stirrer at full speed for about 10 minutes. No heating was required at this stage.
6) Then, the beaker was placed in an Ultrasound Water Bath. No heating was required at this stage.
7) For every 30 minutes, each beaker will be removed from the Ultrasound Water Bath to determine its viscosity using Viscometer
8) After testing, the solution was kept in vials for further use.
9) The results from Viscometer for each shear rate were recorded in the table below.

Trial	**Time (hr)**	**Torque (%)**	**Viscosity (mPa.s)**	**Str Rate (N/m^2)**	**Shear Rate (1/sec)**
1	0.5				
2	1.0				
3	1.5				
4	2.0				
5	2.5				

3.5 To characterize the composite using various analysis such as Viscometer, FT-IR spectral analysis, Thermal Gravimetric Analysis (TGA), and Tensile Test

In this section, the composites will be prepared and characterized for various analyses.

3.5.1 Preparation of Sample for Characterization

1) Carbon Nanotube (CNT) powder was dried for one hour in an preheated oven at 105°C to remove any moisture content in it.
2) A sample with a total volume of 200mL was prepared. The sample was prepared by weight percentage of 0.4% CNT and 99.6% of Polyester Resin.
3) Both the measured CNT and Polyester Resin were added into a 500mL beaker. In addition, Tetrahydrofuran (THF) which acts as a solvent was added into the beaker (Ratio of CNT to THF is 1:25). The mixture was stirred using glass rod for 1-2 minutes.
4) After that, the mixture was stirred using magnetic stirrer at full speed for about 10 minutes. No heating was required at this stage.
5) Then, the beaker was placed in an Ultrasound Water Bath for 1.5 hour. No heating was required at this stage.
6) After 1.5 hour, the beaker was removed from the Ultrasound Water Bath.
7) Then, the beaker as heated to 66°C for 1-2 minutes to remove Tetrahydrofuran from the mixture.
8) The beaker was placed in a water bath to cool the mixture to room temperature.

9) After that, Methyl Ethyl Ketone Peroxide (MEKP) was added to the mixture (Ratio of MEKP to composite is 1:100). The solution was stirred for 1-2 minutes before pouring into the mould to be hardened.
10) After two days, the liquid resin turned into a solid composite. The solid composite is now ready for testing and characterization purpose.

Figure 3.6: Dried CNT is measured

Figure 3.7: CNT is dissolved in THF

Figure 3.8: Sample were stirred using magnetic stirrer

Figure 3.9: Sample is sonicated in Ultrasound Water Bath

Figure 3.10: Sample is stirred after adding MEKP

Figure 3.11: Pure Liquid Resin in Metal Mold

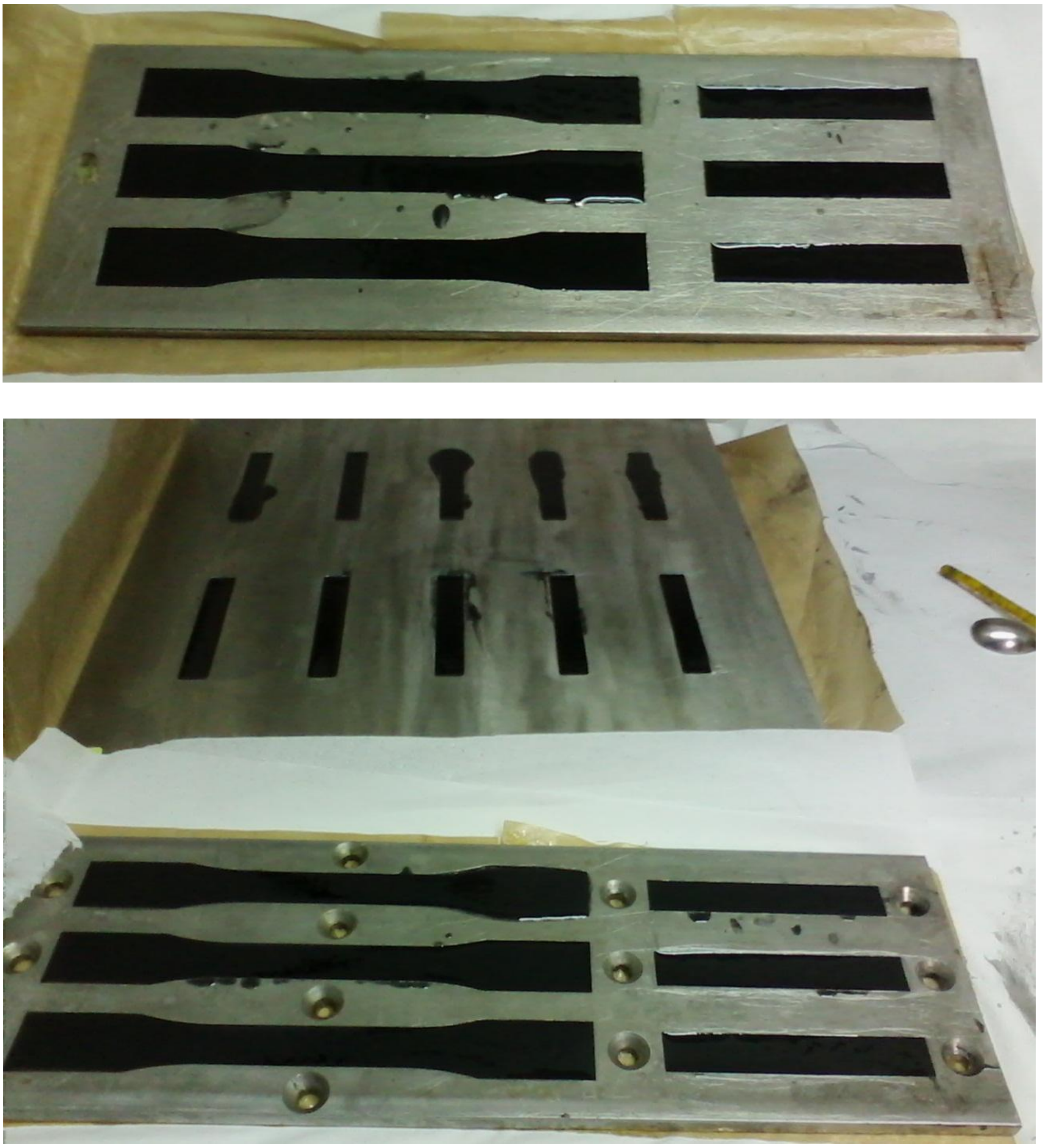


Figure 3.12: Liquid Resin + MWCNT in Metal Mold

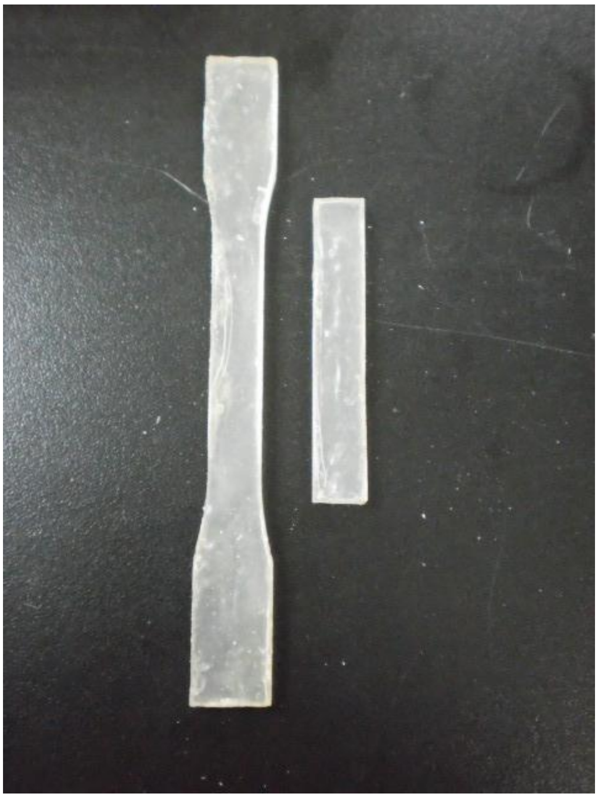


Figure 3.13: Hardened Pure Resin

Figure 3.14: Hardened resin + MWCNT mixture

CHAPTER 4

RESULT & DISCUSSION

4.1 Introduction

In this chapter, all the results obtained are showed clearly in the form of figures whereas the raw data are attached in the Appendix section. The results that will be discussed in this chapter include comparison of viscosity for different weight percentage of CNT when no solvent is used, comparison of viscosity for different weight percentage of CNT when solvent is used, combined comparison of viscosity for different weight percentage of CNT, comparison of sonication time, TGA analysis, tensile test and etc. For the entire test that was performed, average values were calculated for consistent results.

4.2 Optimization

4.2.1 Optimizing Weight Percentage of CNT with No Solvent

Figure 4.1 shows the average viscosity versus shear rate for different weight percentage of CNT (0.1wt % to 0.5wt %) when no solvent was used.

There are many discussions that can be made from this graph. Firstly, the average viscosity of the composite is dependent on the shear rate. For example, when the shear rate is zero, the average viscosity is also zero. However, when the shear rate increases, the average viscosity decreases. Hence, it can be concluded that this composite is a Non-Newtonian fluid.

The overall average viscosity increases when there is an increase in the weight percentage of MWCNT. The average viscosities of 0.1-0.5% MWCNT against shear rate (1/sec) were plotted. Hence, the optimum weight percentage or critical weight percentage of MWCNT is determined as 0.4wt %. According to Shu-quan et al. (2007), beyond a critical weight percentage, the CNTs have a deteriorating effect on the strength and modulus of the composite. This is the main reason for the decrease in the average viscosity beyond the critical weight percentage of MWCNT.

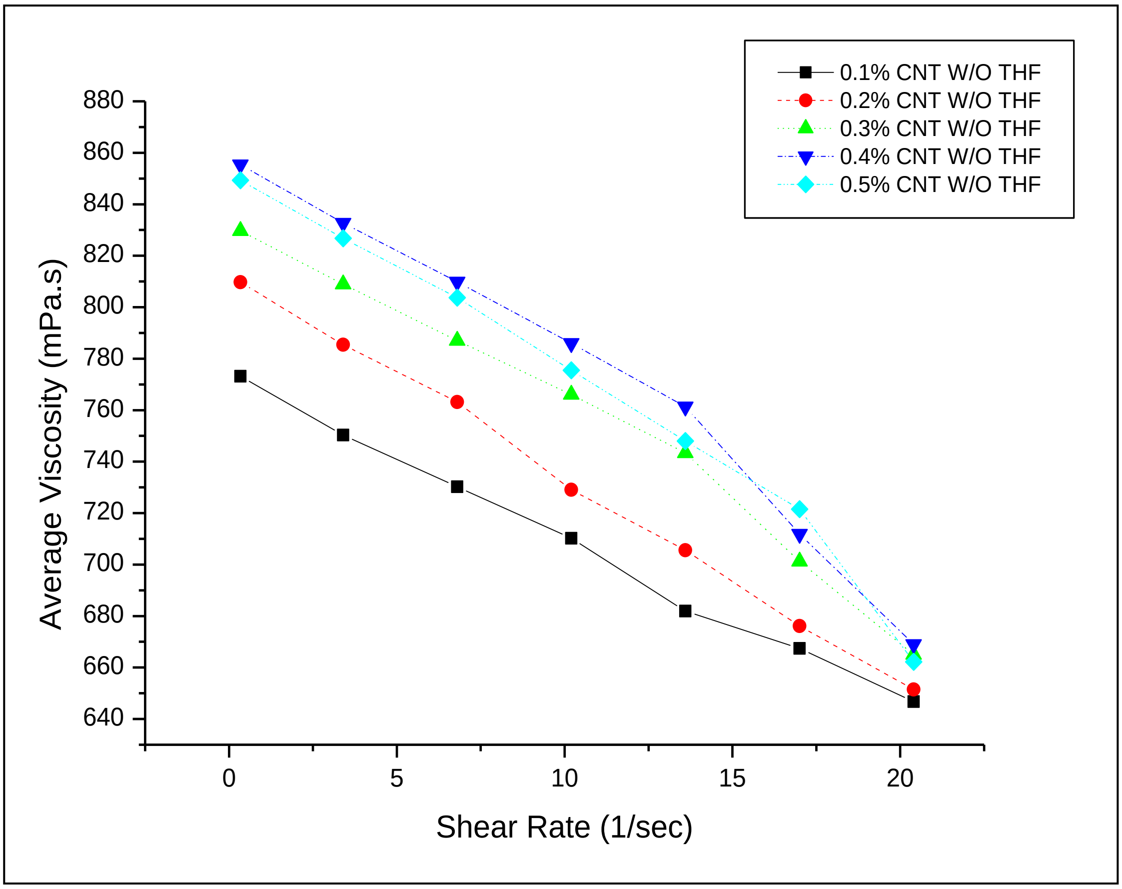


Figure 4.1: Average Viscosity versus Shear Rate for Different Weight Percentage of MWCNT (0.1wt % to 0.5wt %) with No Solvent

4.2.2 Optimizing Weight Percentage of CNT with Solvent

Figure 4.2 shows the average viscosity versus shear rate for different weight percentage of MWCNT (0.1wt % to 0.5wt %) when solvent was used. Generally, the discussions that can be made from the graph below are actually similar to the previous graph when no solvent was used. Firstly, the average viscosity of the composite is dependent to the shear rate. When the shear rate increase, the average viscosity also increase.

When the weight percentage of MWCNT increases from 0.1wt % to 0.6wt %, the overall average viscosity also increases. The average viscosities of 0.1-0.5% MWCNT against shear rate (1/sec) were plotted. 0.4% CNT line shows a higher peak compared to other weight percentages. Hence, the optimum weight percentage or critical weight percentage of this result will be 0.4wt % MWCNT.

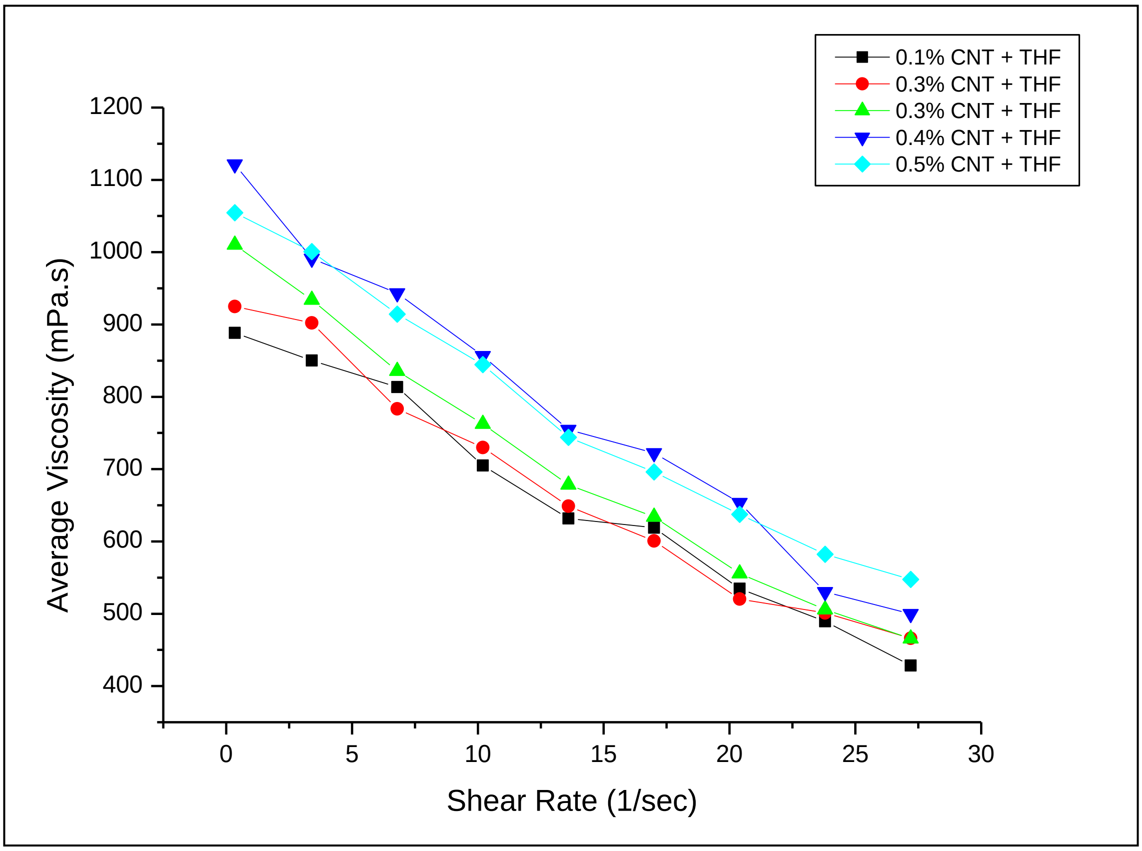


Figure 4.2: Average Viscosity versus Shear Rate for Different Weight Percentage of MWCNT (0.1wt % to 0.5wt %) with Solvent

4.2.3 Comparison of Optimizing Weight Percentage of MWCNT for both without solvent and with solvent

Figure 4.3 shows the comparison of average viscosity versus shear rate for different weight percentage of MWCNT (0.1wt % to 0.5wt %) when both without solvent and with solvent. From the figure, the overall average viscosity when solvent was used is relatively higher compared to the average viscosity when no solvent was used.

From Figure 4.1, the average viscosity is the highest for 0.4wt % when no solvent was used. However, when the average viscosity is compared in this figure, the average viscosity of 0.4wt % is even lower than the average viscosity of 0.1wt % when solvent was used. Hence, we know that by using solvent such as THF, we could raise the average viscosity, and also increase the dispersion of MWCNT in Polyester Resin.

As a conclusion, the weight percentage of MWCNT has been optimized which is 0.4wt % with the usage of solvent. In the following section, this optimization will be further improved by optimizing the sonication time of the composite.

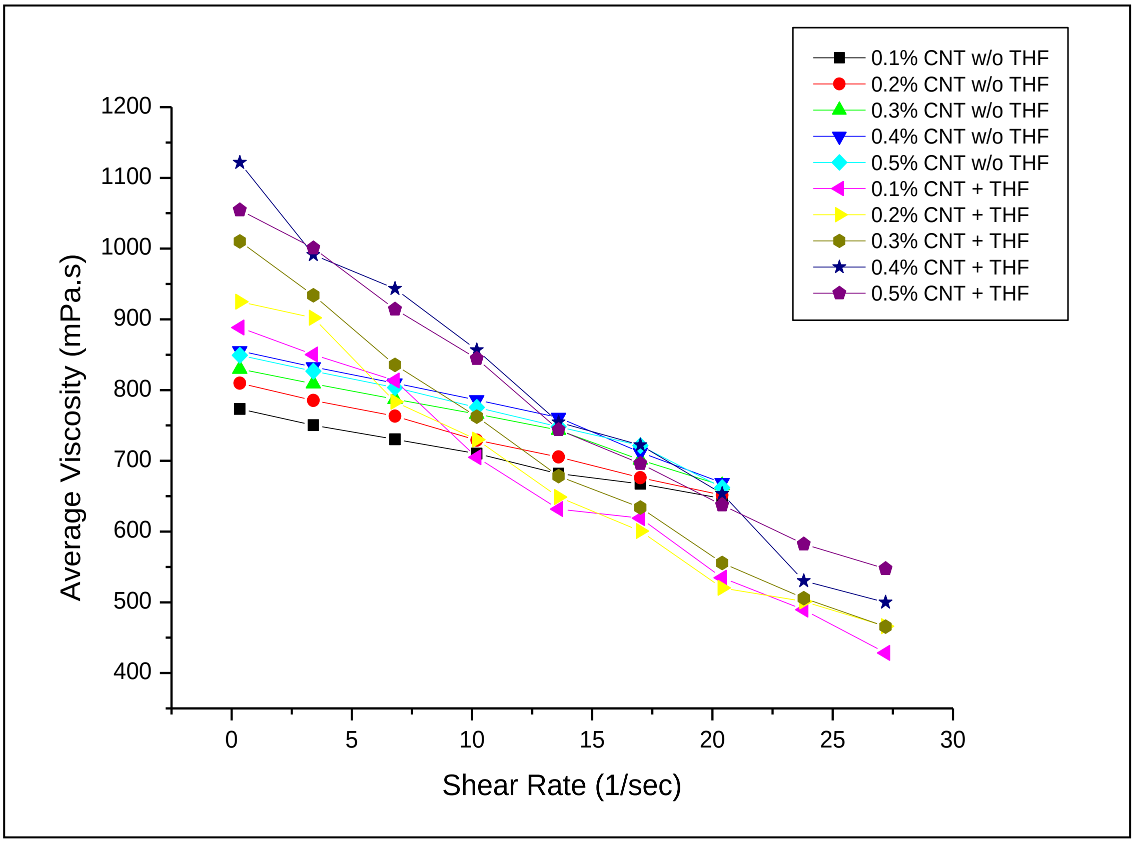


Figure 4.3: Comparison of Average Viscosity versus Shear Rate for Different Weight Percentage of MWCNT (0.1wt % to 0.5wt %)

4.2.4 Optimizing the Sonication Time

Figure 4.4 shows the average viscosity versus shear rate for different sonication time. To obtain this result, weight percentage of 0.4% with solvent was chosen. There are some notable discussions that can be made from the figure below. The first discussions are similar to the previous figures whereby the viscosity reduces when the shear rate increases.

The average viscosity is very low after sonication of 30 minutes. This is because the MWCNT has not fully dispersed in the Polyester Resin. As we know earlier, that when the viscosity is lower, the dispersion is not good. However, after 30 minutes, the average viscosity increases drastically until 90 minutes. The average viscosity is the highest after 90 minutes of sonication. Beyond 90 minutes of sonication, there is very small increment in the average viscosity of the composite. This is because after a point in the sonication time, the dispersion of MWCNT is at maximum. Hence, further increase in the sonication time will not improve the dispersion of MWCNT. In addition, waiting for a long time just to sonicate the composite for small changes in average viscosity is not practically feasible. Hence, the optimum sonication time chosen for this study will be 90 minutes.

As a conclusion, the weight percentage of MWCNT has been optimized which is 0.4wt % with the usage of solvent whereas the sonication time was optimized to 90 minutes. In the following section, the optimization will be applied to the composite and will be thoroughly characterized. The result of the characterization will be explained in the next section.

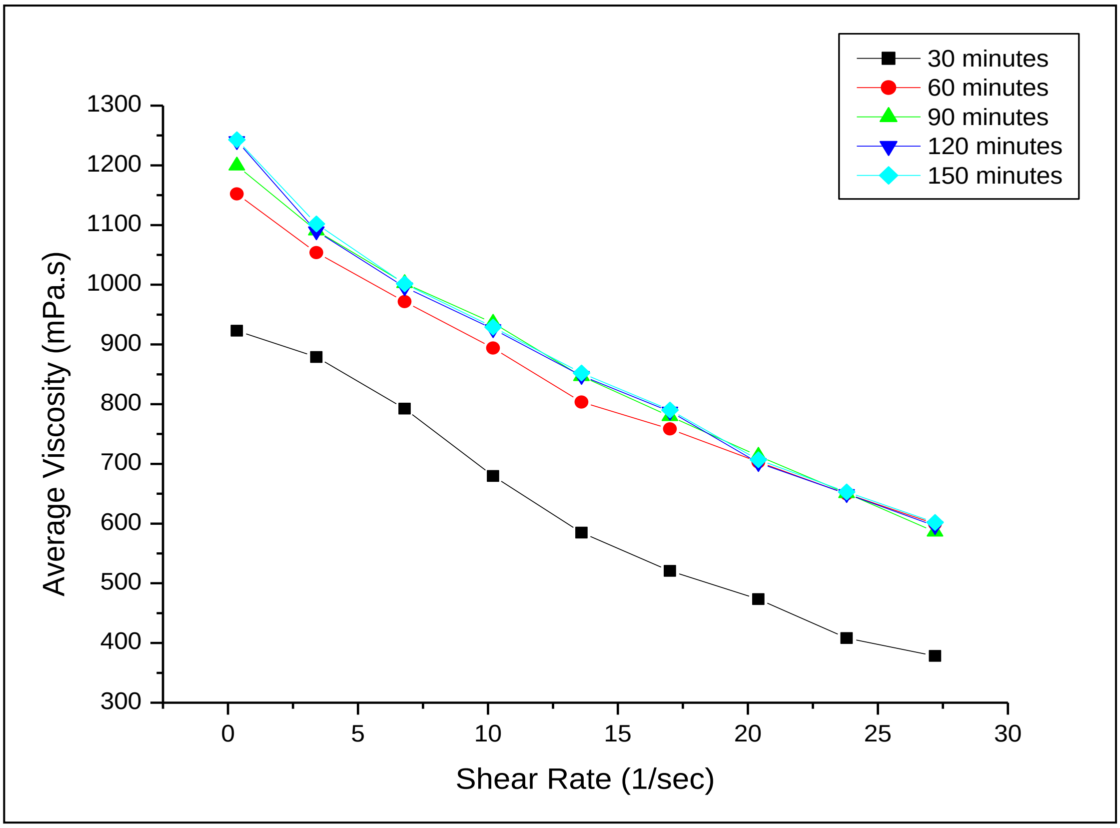


Figure 4.4: Average Viscosity versus Shear Rate for Different Sonication Time

4.3 Characterization

4.3.1 Thermal Gravimetric Analysis (TGA)

Thermal (TGA) studies of samples were carried-out in a compressed air atmosphere with 60:40 ratio on a thermal analyser at a heating rate of 10 ºC /min. The objective of thermal analysis is to study the effect of heating on the materials so that the stability of the materials at elevated temperature could be known for its applications in various fields. The results of thermal degradation are shown in Figure 4.5. Pure polyester resin samples showed considerable weight loss in comparison to the samples that have been treated with MWCNTs. Polyester resins that treated MWCNTs had a lower initial temperature of thermal degradation. The polyester resin that mixed with 0.4 wt% MWCNTs have less thermal degradation due to higher thermal stability. Thermal stability causes the boards treated with 0.4 wt % MWCNTs + Polyester resin decreases thermal degradation of the samples. Generally, at the higher temperature, condensable vapors were produced. At last, by increasing the MWCNTs percentage the thermal stability was enhanced in the Polyester resin.

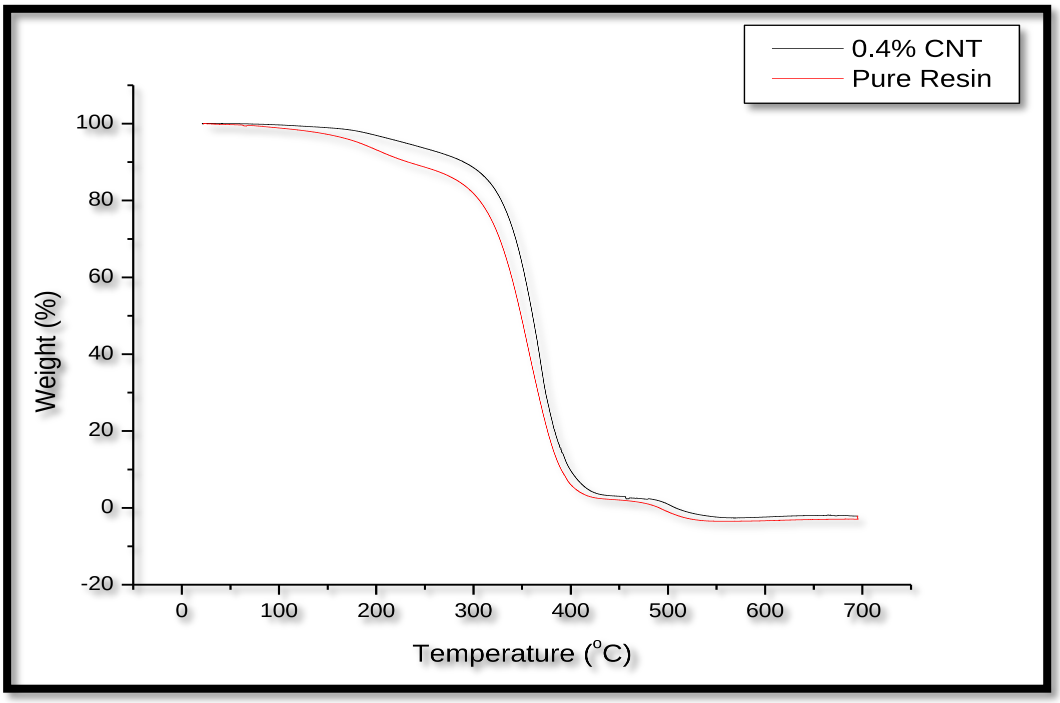


Figure 4.5: Comparison of TGA results for Pure Resin and 0.4% CNT

4.3.2 Tensile Test

Tensile testing had been conducted on pure polyester resin samples and 0.4% CNT + polyester composite samples. This test is performed by using Universal Testing Machine (UTM). The crosshead speed is fixed at a rate of 3mm/min. The shape of the samples used for this testing is "Dumbell" shape. Average Tensile strength for 0.4% CNT is 2525.45 N and for Pure Polyester resin is 1176 N. From Table 4.1, we can conclude that this research study is successful because the main objective of this study is achieved. The maximum force, maximum stress and maximum strain for 0.4% CNT had been increased two times compared to pure polyester resin. This is because Multi-walled Carbon Nanotube (MWCNT) has a higher mechanical strength. I had increased the strength of pure polyester resin by adding optimum weight percentage of carbon nanotube, 0.4% and optimum sonication time at 90 minutes.

Name Parameter Unit	Max.Force Calc (N)	Max.Stress Calc (N/mm^2)	Max.Strain Calc (%)
Pure Resin 1	959.040	19.9800	2.90133
Pure Resin 2	1087.97	22.6659	3.00948
Pure Resin 3	1480.98	30.8538	4.63399
Average	**1176.00**	**24.4999**	**3.51493**
Standard Deviation	**271.877**	**5.66415**	**0.77651**
0.4% CNT	2689.39	56.0290	7.14427
0.4% CNT	1493.98	31.1246	5.03695
0.4% CNT	3392.98	70.6870	9.14797
Average	**2525.45**	**52.6135**	**7.10973**
Standard Deviation	**960.056**	**20.0011**	**1.64458**

Table 4.1 Tensile test data for Pure Polyester Resin & 0.4% CNT + Polyester composite

4.3.3 Fourier Transform Infrared Spectroscopy (FTIR)

From the Figure 4.7, it was generally observed that polyester groups present at 1500 cm^{-1} in the spectrum of all the pure polyester samples. Recent research on unsaturated polyester resin by Sudirman.et.al (2012) states that polyester groups present at 1596 cm^{-1} from FTIR analysis. Around the frequency of 3830 cm^{-1}-3150cm^{-1} free OH stretching of alcohol and phenol groups were identified. From Figure 4.8, FTIR analysis of 0.4wt % MWCNT + Polyester resin shows dominant at 3760, 3580, 2360, 970 cm^{-1} which corresponds to, C-N, C-H, CNT, C-O and C-C respectively. The covalent linkage between the MWCNT and functional group in polyester resin was identified. In Figure 4.9, a comparison graph was drawn for pure polyester resin and 0.4% CNT + polyester composites. The attachments of MWCNTs to functional groups significantly improve their properties. For more assurance, a wavelength and frequency range of polymers were referred to identify the chemical groups present in the samples. The diagram is inserted below, Figure 4.6.

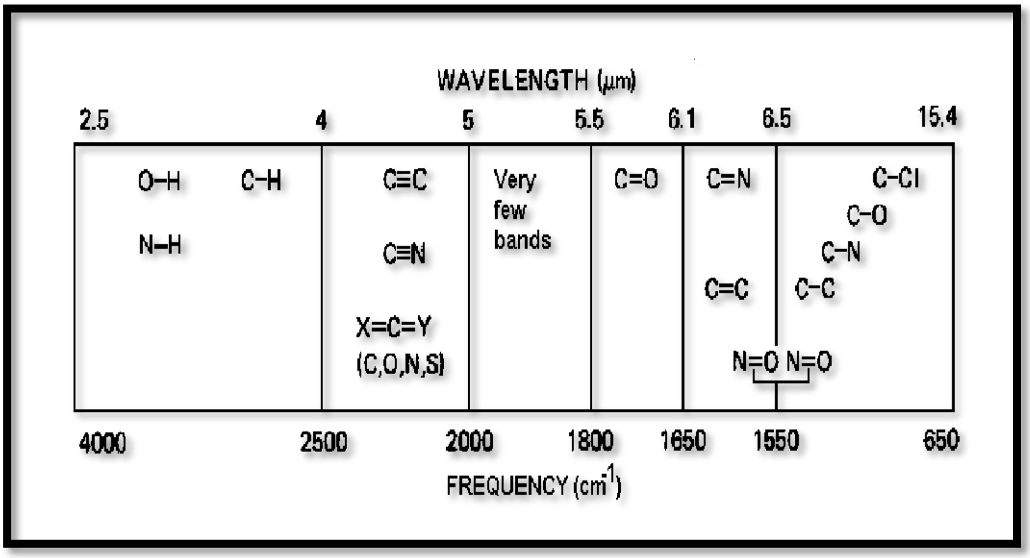


Figure 4.6: Wavelength & frequency range of polymers

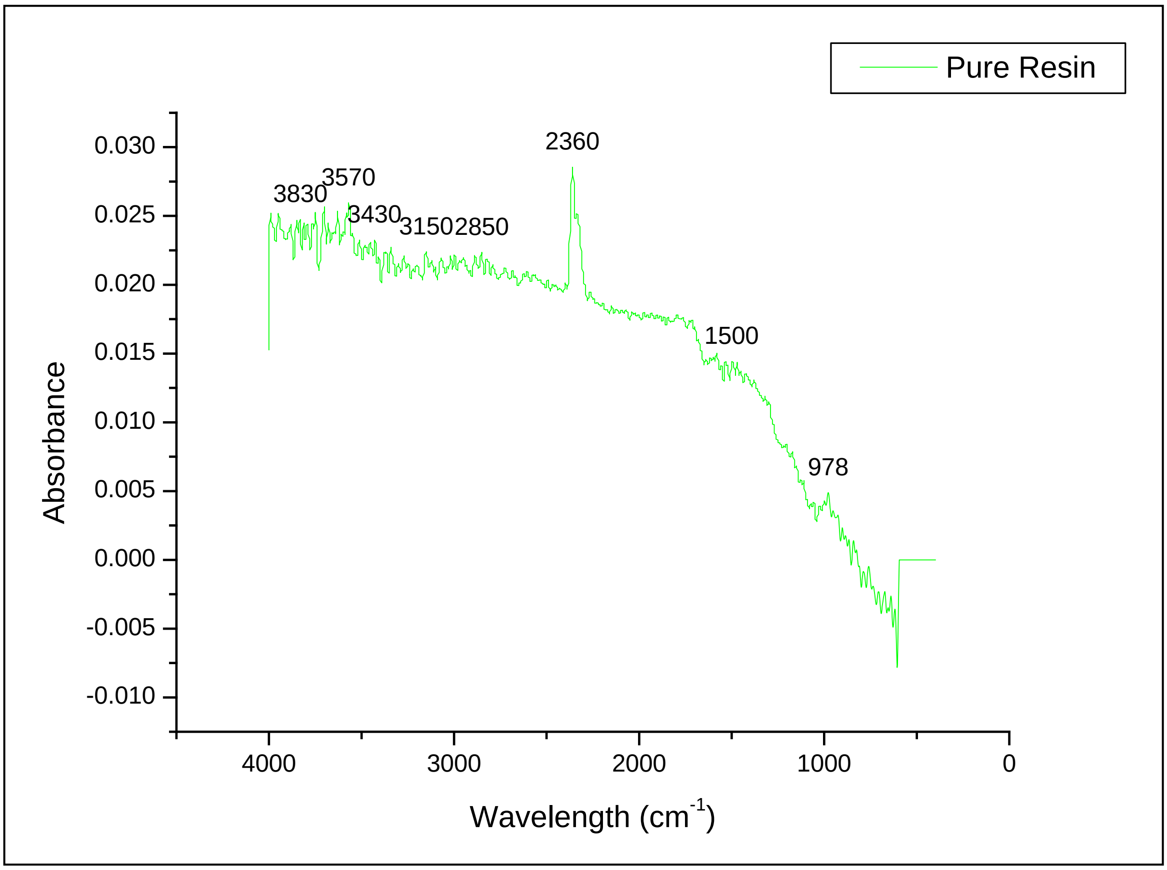


Figure 4.7: FTIR analysis for Pure Polyester Resin

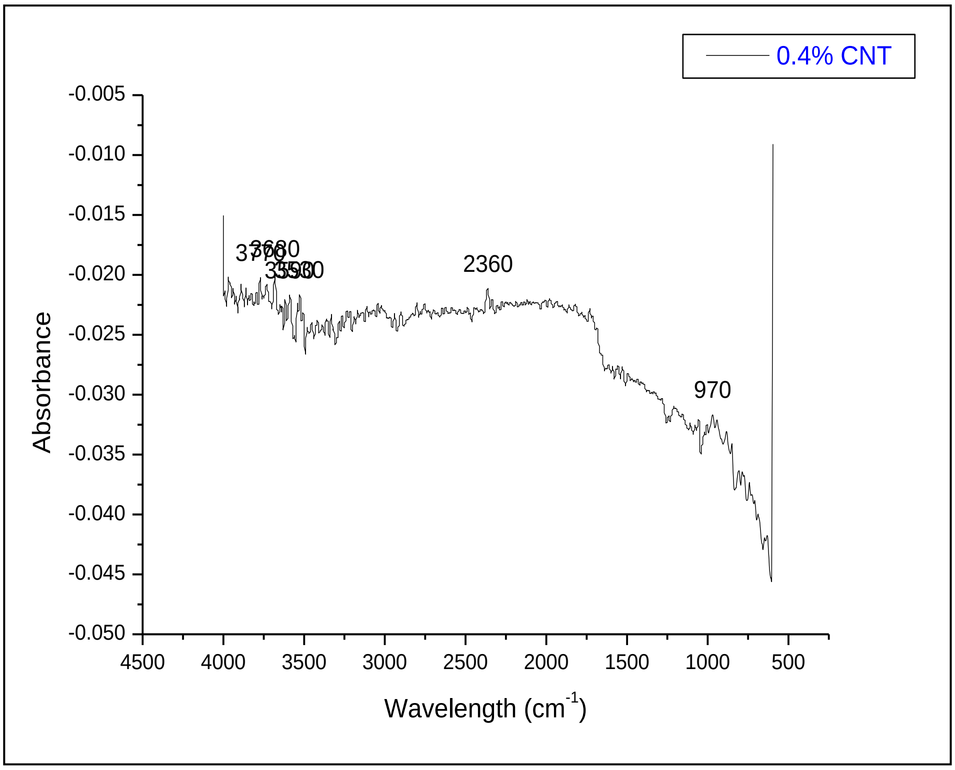


Figure 4.8: FTIR Analysis for 0.4% CNT

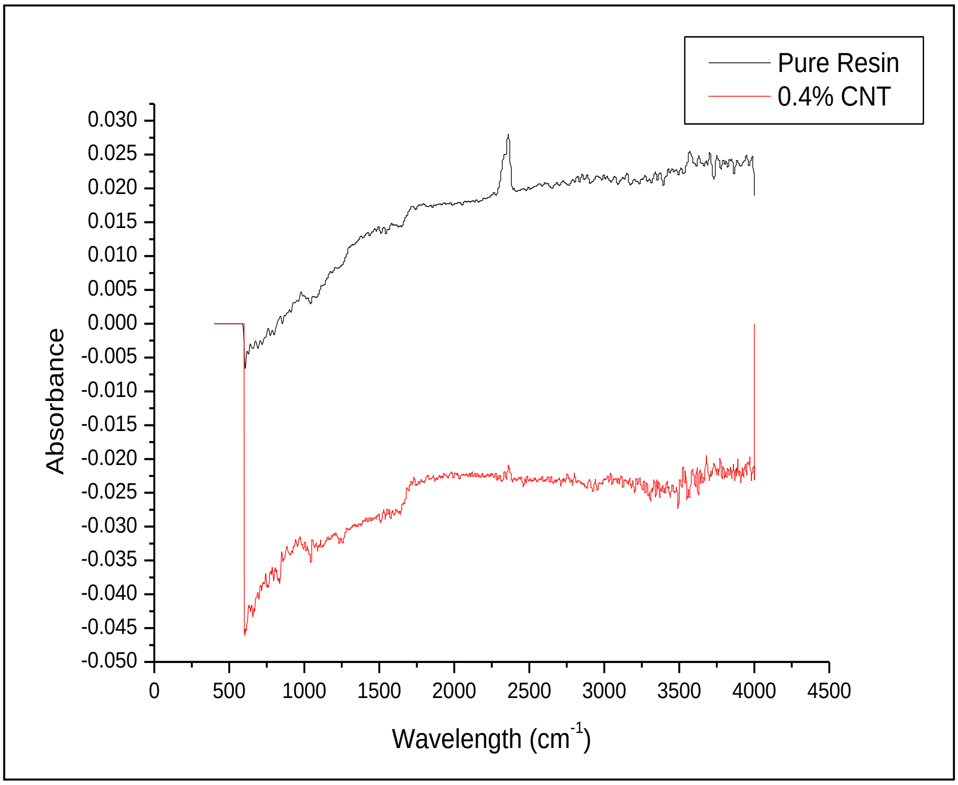


Figure 4.9: Comparison of FTIR analyses for Pure Resin & 0.4% CNT

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.1 Conclusion

This project focuses on optimization of carbon nanotube weight percentage, 0.1-0.5% for both using solvent and without solvent. It is identified that 0.4wt% of Carbon Nanotube (CNT) using solvent, THF gives a higher viscosity compared to samples without using solvent. Then, the optimum sonication time is 90 minutes. Tensile strength for 0.4% CNT is 2525.45 N compared to 1176 N for pure polyester resin. From Thermal Gravimetric Analysis (TGA), can be concluded that the polyester resin that mixed with 0.4 wt% MWCNTs have less thermal degradation due to higher thermal stability. FTIR analysis of 0.4wt % MWCNT + Polyester resin shows dominant at 3760, 3580, 2360, 970 cm^{-1} which corresponds to, C-N, C-H, CNT, C-O and C-C respectively.

5.2 Recommendations

This research work can be expanded by adding the parameters needed to be experimented. For example, in this research, I used Tetrahydrofuran (THF) as the solvent for carbon nanotube. But for more accurate reading, we can add the parameter of choice of solvent for this research work. As we know, there are a lot of solvents used in industry for carbon nanotube. Thus, by doing this I believe can improve the results obtained.

5.3 Future work

The research carried is currently being expanded by Mr. A.K.M Moshiul Alam (a Phd student) by expanding the scope of this research. He had been testing the strength of different groups of carbon nanotube mixture with polyester resin. He is comparing the strength of Carboxyl-group carbon nanoutbe, Hydroxyl-group carbon nanotube, and None-functionalized carbon nanotube.

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APPENDIX A: Results for Optimizing CNT wt % for No Solvent

a) 0.1% CNT (0.05 g CNT)

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.10	0.00	0.02	0.40	0.00	0.05	0.20	0.00	0.03	0.23	0.00	0.03	0.00
1.00	2.08	731.24	0.21	2.19	711.23	0.22	1.99	727.05	0.21	2.09	773.20	0.21	0.34
10.00	20.80	694.31	2.14	21.90	675.45	2.16	19.90	703.45	2.11	20.87	750.34	2.14	3.40
20.00	38.30	675.07	3.86	38.40	652.85	3.89	35.50	674.90	3.81	37.40	730.26	3.85	6.80
30.00	54.00	649.23	5.44	54.90	620.34	5.48	52.90	644.97	5.39	53.93	710.22	5.44	10.20
40.00	68.80	621.50	6.99	69.10	601.70	7.02	64.90	620.49	6.56	67.60	681.90	6.86	13.60
50.00	83.30	599.63	8.47	89.90	582.30	8.44	81.90	602.43	8.44	85.03	667.45	8.45	17.00
60.00	95.80	581.31	9.95	97.60	565.09	9.90	92.30	582.03	9.90	95.23	646.75	9.92	20.40
70.00		-	-	-	-	-	99.70	563.30	10.10	-	-	-	23.80
80.00	-	-	-	-	-	-	-	-	-	-	-	-	27.20

b) 0.2% CNT (0.10 g CNT)

RPM	Trial 1			Trial 2			Trial 3			**Average**			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.20	0.00	0.02	0.10	0.00	0.01	0.30	0.00	0.02	0.20	0.00	0.02	0.00
1.00	2.07	790.22	0.22	1.99	821.32	0.21	2.13	817.65	0.22	2.06	809.73	0.21	0.34
10.00	20.70	771.03	2.15	19.90	799.34	2.10	21.30	785.90	2.17	20.63	785.42	2.14	3.40
20.00	38.20	750.35	3.85	37.60	773.68	3.81	39.00	765.46	3.88	38.27	763.16	3.85	6.80
30.00	54.20	721.04	5.49	54.10	730.83	5.44	55.60	735.24	5.51	54.63	729.04	5.48	10.20
40.00	69.50	684.56	7.02	69.00	712.32	6.99	73.90	719.82	7.05	70.80	705.57	7.02	13.60
50.00	84.10	667.34	8.54	82.90	674.53	8.52	88.10	686.59	8.58	85.03	676.15	8.55	17.00
60.00	98.80	636.57	10.10	97.50	653.24	10.00	99.90	664.65	10.10	98.73	651.49	10.07	20.40
70.00	-	-	-	-	-	-	-	-	-	-	-	-	23.80
80.00	-	-	-	-	-	-	-	-	-	-	-	-	27.20

c) 0.3% CNT (0.15 g CNT)

RPM	Trial 1			Trial 2			Trial 3			**Average**			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.50	0.00	0.05	0.10	0.00	0.01	0.20	0.00	0.01	0.27	0.00	0.02	0.00
1.00	2.14	863.32	0.23	2.09	823.45	0.22	2.22	802.31	0.23	2.15	829.69	0.22	0.34
10.00	21.40	843.12	2.25	20.90	796.09	2.21	22.20	787.60	2.25	21.50	808.94	2.24	3.40
20.00	40.20	820.72	4.07	39.80	775.34	4.05	43.30	765.09	4.09	41.10	787.05	4.07	6.80
30.00	56.90	799.43	5.74	55.50	749.80	5.73	58.90	748.99	5.75	57.10	766.07	5.74	10.20
40.00	72.80	781.10	7.37	71.10	725.46	7.36	74.90	723.42	7.39	72.93	743.33	7.37	13.60
50.00	88.20	763.42	8.95	88.00	709.83	8.92	91.10	695.86	8.99	89.10	701.20	8.95	17.00
60.00	98.10	738.28	9.92	97.20	688.23	9.90	99.90	630.23	10.00	98.40	665.03	9.94	20.40
70.00	-	-	-	-	-	-	-	-	-	-	-	-	23.80
80.00	-	-	-	-	-	-	-	-	-	-	-	-	27.20

d) 0.4% CNT (0.20 g CNT)

RPM	Trial 1			Trial 2			Trial 3			**Average**			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.10	0.00	0.02	0.20	0.00	0.01	0.50	0.00	0.05	0.27	0.00	0.03	0.00
1.00	2.27	893.43	0.24	2.25	874.35	0.23	2.11	798.54	0.23	2.21	855.44	0.23	0.34
10.00	22.70	874.35	2.35	22.50	856.09	2.34	21.10	767.77	2.33	22.10	832.74	2.34	3.40
20.00	41.10	846.86	4.17	43.90	832.90	4.19	40.90	749.83	4.14	41.97	809.86	4.17	6.80
30.00	57.80	812.80	5.84	59.10	821.78	5.99	56.30	723.54	5.81	57.73	786.04	5.88	10.20
40.00	73.40	796.56	7.40	74.70	795.32	7.45	69.90	692.04	7.29	72.67	761.31	7.38	13.60
50.00	88.20	774.54	8.92	89.10	763.86	9.04	80.90	660.05	8.67	86.07	711.90	8.88	17.00
60.00	99.30	753.09	10.10	99.90	743.82	10.10	89.90	643.21	9.15	96.37	669.05	9.78	20.40
70.00	-	-	-	-	-	-	99.80	614.35	10.10	-	-	-	23.80
80.00	-	-	-	-	-	-	-	-	-	-	-	-	27.20

e) 0.5% CNT (0.25 g CNT)

RPM	Trial 1			Trial 2			Trial 3			**Average**			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.50	0.00	0.05	0.10	0.00	0.01	0.10	0.00	0.01	0.23	0.00	0.02	0.00
1.00	2.28	875.63	0.24	2.31	862.23	0.24	2.41	810.10	0.24	2.33	849.32	0.24	0.34
10.00	22.80	862.31	2.36	23.10	834.53	2.39	24.10	783.42	2.41	23.33	826.75	2.39	3.40
20.00	42.20	845.32	4.26	43.30	802.31	4.29	44.40	763.43	4.31	43.30	803.69	4.29	6.80
30.00	59.60	823.42	6.02	60.90	772.19	6.15	61.90	730.83	6.19	60.80	775.48	6.12	10.20
40.00	76.00	790.43	7.70	79.90	743.32	7.75	83.90	710.34	7.78	79.93	748.03	7.74	13.60
50.00	88.90	773.24	9.30	89.90	721.05	9.33	90.10	670.32	9.95	89.63	721.54	9.53	17.00
60.00	98.30	756.43	10.00	98.90	620.12	10.10	99.90	610.02	10.10	99.03	662.19	10.07	20.40
70.00	-	-	-	-	-	-	-	-	-	-	-	-	23.80
80.00	-	-	-	-	-	-	-	-	-	-	-	-	27.20

APPENDIX B: Results for Optimizing CNT wt % With THF

a) 0.1% CNT (0.05 g CNT; 1.25 mL THF)

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.10	0.00	0.01	0.50	0.00	0.05	0.20	0.00	0.01	0.27	0.00	0.02	0.00
1.00	1.87	889.65	0.21	1.92	918.46	0.22	1.72	857.36	0.20	1.84	888.49	0.21	0.34
10.00	18.70	862.10	2.09	19.20	899.32	2.15	17.20	852.21	2.03	18.37	850.23	2.09	3.40
20.00	29.80	780.64	2.98	30.10	821.82	3.02	28.30	800.23	2.93	29.40	813.42	2.98	6.80
30.00	38.50	698.90	3.89	39.00	720.20	3.91	36.50	695.50	3.85	38.00	704.87	3.88	10.20
40.00	45.50	642.20	4.56	47.60	613.30	4.71	44.10	639.90	4.52	45.73	631.80	4.60	13.60
50.00	59.90	594.84	6.01	62.10	619.90	6.23	57.50	642.21	5.95	59.83	618.98	6.06	17.00
60.00	71.20	439.90	7.02	72.90	521.30	7.05	69.90	477.70	6.99	71.33	534.73	7.02	20.40
70.00	88.30	447.20	8.96	89.90	483.90	9.01	86.50	410.10	8.91	88.23	489.54	8.96	23.80
80.00	96.70	419.10	9.96	99.80	443.20	10.10	95.50	423.21	9.93	97.33	428.50	10.00	27.20

b) 0.2% CNT (0.10 g CNT; 2.50 mL THF)

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.20	0.00	0.01	0.10	0.00	0.01	0.20	0.00	0.01	0.17	0.00	0.01	0.00
1.00	2.13	921.69	0.22	2.15	964.26	0.22	2.11	888.59	0.22	2.13	924.85	0.22	0.34
10.00	21.30	899.90	2.18	21.50	930.90	2.19	21.10	875.50	2.17	21.30	902.10	2.18	3.40
20.00	30.50	787.70	3.04	32.20	829.90	3.09	29.80	732.20	3.02	30.83	783.27	3.05	6.80
30.00	37.90	729.90	3.86	41.10	759.50	4.01	36.50	699.90	3.83	38.50	729.77	3.90	10.20
40.00	47.90	635.50	4.73	52.00	688.80	5.23	46.70	622.20	4.71	48.87	648.83	4.89	13.60
50.00	62.00	599.90	6.23	65.30	634.50	6.55	61.10	567.70	6.21	62.80	600.70	6.33	17.00
60.00	71.10	543.30	7.01	74.40	525.50	7.15	73.30	491.90	6.99	72.93	520.23	7.05	20.40
70.00	90.00	517.90	9.02	89.90	509.90	9.01	88.20	475.50	8.77	89.37	501.10	8.93	23.80
80.00	98.80	486.50	9.95	99.90	468.60	10.10	97.60	443.20	9.89	98.77	466.10	9.98	27.20

c) 0.3% CNT (0.15 g CNT; 3.75 mL THF)

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.20	0.00	0.01	0.10	0.00	0.01	0.10	0.00	0.01	0.13	0.00	0.01	0.00
1.00	1.71	995.68	0.20	2.14	1010.10	0.22	1.88	960.20	0.21	1.91	1010.21	0.21	0.34
10.00	17.10	931.90	2.03	21.40	969.90	2.19	18.80	899.90	2.09	19.10	933.90	2.10	3.40
20.00	28.10	833.30	2.95	32.30	872.20	3.09	29.90	801.10	2.98	30.10	835.53	3.01	6.80
30.00	36.40	765.50	3.86	40.20	799.90	4.01	38.50	721.90	3.89	38.37	762.43	3.92	10.20
40.00	44.40	688.80	4.52	52.20	723.30	5.23	45.60	623.30	4.56	47.40	678.47	4.77	13.60
50.00	57.80	634.40	5.95	66.50	667.90	6.55	59.90	599.90	6.01	61.40	634.07	6.17	17.00
60.00	70.00	556.60	7.01	74.50	587.00	7.15	71.20	523.30	7.02	71.90	555.63	7.06	20.40
70.00	87.10	499.90	8.92	89.90	538.80	9.01	88.40	478.90	8.96	88.47	505.87	8.96	23.80
80.00	96.20	465.50	9.95	99.80	499.90	10.10	96.70	431.10	9.92	97.57	465.50	9.99	27.20

d) 0.4% CNT (0.20 g CNT; 5.00 mL THF)

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.20	0.00	0.01	0.10	0.00	0.01	0.20	0.00	0.01	0.17	0.00	0.01	0.00
1.00	1.69	1176.45	0.20	2.15	1085.03	0.22	2.13	1103.43	0.22	1.99	1121.64	0.21	0.34
10.00	16.90	987.70	2.02	21.50	984.40	2.19	21.30	972.20	2.18	19.90	991.21	2.13	3.40
20.00	28.50	875.50	2.93	32.20	953.42	3.09	30.50	879.03	3.04	30.40	943.42	3.02	6.80
30.00	36.30	799.90	3.85	41.10	833.30	4.01	37.90	767.90	3.86	38.43	856.54	3.91	10.20
40.00	43.90	712.60	4.53	52.00	735.60	5.23	47.90	687.70	4.73	47.93	754.43	4.83	13.60
50.00	57.20	689.90	5.95	65.30	704.40	6.55	62.00	649.90	6.23	61.50	722.34	6.24	17.00
60.00	70.10	589.90	7.00	74.40	654.40	7.15	71.10	565.50	7.01	71.87	653.43	7.05	20.40
70.00	87.70	534.40	8.92	89.90	589.90	9.01	90.00	499.90	9.02	89.20	530.24	8.98	23.80
80.00	97.40	499.90	10.00	99.90	534.30	10.10	98.80	465.50	9.95	98.70	499.90	10.02	27.20

e) 0.5% CNT (0.25 g CNT; 6.25 mL THF)

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.20	0.00	0.01	0.50	0.00	0.05	0.20	0.00	0.01	0.30	0.00	0.02	0.00
1.00	1.81	1039.43	0.21	1.91	1070.23	0.21	1.51	1012.51	0.20	1.74	1054.45	0.20	0.34
10.00	18.10	999.90	2.05	19.10	1032.90	2.08	15.10	969.90	1.97	17.43	1000.90	2.03	3.40
20.00	29.60	912.20	2.97	29.90	943.30	2.98	26.60	887.70	2.88	28.70	914.40	2.94	6.80
30.00	37.80	844.40	3.86	38.30	876.60	3.89	33.30	812.20	3.78	36.47	844.40	3.84	10.20
40.00	44.50	740.90	4.54	48.80	778.80	4.61	45.50	711.90	4.48	46.27	743.87	4.54	13.60
50.00	58.80	699.40	5.99	63.30	732.20	6.24	59.90	656.60	6.12	60.67	696.07	6.12	17.00
60.00	70.90	633.30	7.00	72.20	678.90	7.09	70.10	599.90	6.87	71.07	637.37	6.99	20.40
70.00	87.70	587.70	8.92	87.30	612.20	8.91	88.10	546.40	8.93	87.70	582.10	8.92	23.80
80.00	98.80	543.30	10.00	99.80	587.70	10.10	97.60	511.10	9.92	98.73	547.37	10.01	27.20

APPENDIX C: Overall Comparison for Optimum Weight Percentage of CNT

S Rate (1/sec)	Viscosity (mPa.s)									
	No Solvent					With Solvent				
	0.1% CNT	0.2% CNT	0.3% CNT	0.4% CNT	0.5% CNT	0.1% CNT	0.2% CNT	0.3% CNT	0.4% CNT	0.5% CNT
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.34	773.20	809.73	829.69	855.44	849.32	888.49	924.85	1010.21	1121.64	1054.45
3.40	750.34	785.42	808.94	832.74	826.75	850.23	902.10	933.90	991.21	1000.90
6.80	730.26	763.16	787.05	809.86	803.69	813.42	783.27	835.53	943.42	914.40
10.20	710.22	729.04	766.07	786.04	775.48	704.87	729.77	762.43	856.54	844.40
13.60	681.90	705.57	743.33	761.31	748.03	631.80	648.83	678.47	754.43	743.87
17.00	667.45	676.15	701.20	711.90	721.54	618.98	600.70	634.07	722.34	696.07
20.40	646.75	651.49	665.03	669.05	662.19	534.73	520.23	555.63	653.43	637.37
23.80						489.54	501.10	505.87	530.24	582.10
27.20						428.50	466.10	465.50	499.90	547.37

APPENDIX D: Results for Optimizing Sonication Time

a) 30 minutes

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.50	0.00	0.05	0.10	0.00	0.01	0.20	0.00	0.02	0.27	0.00	0.03	0.00
1.00	1.99	940.88	0.22	1.87	866.31	0.20	2.01	961.44	0.23	1.96	922.88	0.22	0.34
10.00	19.90	870.44	2.15	18.70	845.50	1.98	20.10	921.10	2.32	19.57	879.01	2.15	3.40
20.00	28.40	799.60	2.99	27.30	734.40	2.88	29.90	843.30	2.90	28.53	792.43	2.92	6.80
30.00	35.40	684.40	3.76	33.30	632.20	3.65	38.70	722.20	3.79	35.80	679.60	3.73	10.20
40.00	42.10	589.90	4.21	41.00	532.20	4.17	45.50	632.20	4.32	42.87	584.77	4.23	13.60
50.00	59.90	541.90	5.55	57.80	476.60	5.51	61.10	543.30	5.87	59.60	520.60	5.64	17.00
60.00	70.90	467.40	7.60	69.10	421.10	7.55	73.20	532.20	7.71	71.07	473.57	7.62	20.40
70.00	88.20	402.10	8.98	87.60	376.60	8.95	89.90	445.50	9.03	88.57	408.07	8.99	23.80
80.00	99.80	368.10	10.00	95.60	343.30	9.87	99.90	423.20	10.10	98.43	378.20	9.99	27.20

b) 60 minutes

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.20	0.00	0.01	0.30	0.00	0.02	0.50	0.00	0.05	0.33	0.00	0.03	0.00
1.00	1.69	1186.33	0.20	1.88	1138.25	0.20	1.91	1131.79	0.21	1.83	1152.12	0.20	0.34
10.00	16.90	1050.90	2.02	18.80	1109.20	2.01	19.10	1001.10	2.08	18.27	1053.73	2.04	3.40
20.00	28.50	974.40	2.93	30.10	999.20	3.29	29.90	941.10	2.98	29.50	971.57	3.07	6.80
30.00	36.30	897.20	3.85	39.90	931.10	3.94	38.30	853.30	3.89	38.17	893.87	3.89	10.20
40.00	43.90	800.90	4.53	48.20	844.40	4.56	48.80	765.50	4.61	46.97	803.60	4.57	13.60
50.00	57.20	744.40	5.95	65.50	776.60	6.55	63.30	754.50	6.24	62.00	758.50	6.25	17.00
60.00	70.10	686.60	7.00	78.90	713.30	7.50	72.20	709.80	7.09	73.73	703.23	7.20	20.40
70.00	87.70	631.10	8.92	89.90	653.30	9.02	87.30	665.30	8.91	88.30	649.90	8.95	23.80
80.00	97.40	589.80	10.00	98.40	609.50	9.89	99.80	599.90	10.10	98.53	599.73	10.00	27.20

c) 90 minutes

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.20	0.00	0.01	0.10	0.00	0.01	0.10	0.00	0.01	0.13	0.00	0.01	0.00
1.00	1.72	1230.34	0.20	2.15	1189.43	0.22	2.15	1178.65	0.22	2.01	1199.47	0.21	0.34
10.00	17.20	1096.60	2.03	21.50	1080.10	2.19	21.50	1095.50	2.19	20.07	1090.73	2.14	3.40
20.00	28.30	1002.20	2.93	32.20	1020.30	3.09	32.20	984.40	3.09	30.90	1002.30	3.04	6.80
30.00	36.50	932.90	3.85	41.10	955.50	4.01	41.10	919.30	4.01	39.57	935.90	3.96	10.20
40.00	44.10	853.30	4.52	52.00	886.60	5.23	52.00	799.90	5.23	49.37	846.60	4.99	13.60
50.00	57.50	786.30	5.95	65.30	799.10	6.55	65.30	753.30	6.55	62.70	779.57	6.35	17.00
60.00	69.90	711.10	6.99	74.40	742.20	7.15	74.40	686.60	7.15	72.90	713.30	7.10	20.40
70.00	86.50	664.40	8.91	89.90	685.40	9.01	89.90	602.20	9.01	88.77	650.67	8.98	23.80
80.00	95.50	593.30	9.93	99.90	612.20	10.10	99.90	553.30	10.10	98.43	586.27	10.04	27.20

d) 120 minutes

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.20	0.00	0.01	0.20	0.00	0.01	0.50	0.00	0.05	0.30	0.00	0.02	0.00
1.00	2.11	1236.23	0.22	2.13	1242.31	0.22	1.92	1243.65	0.22	2.05	1240.73	0.22	0.34
10.00	21.10	1099.60	2.17	21.30	1048.40	2.18	19.20	1121.10	2.15	20.53	1089.70	2.17	3.40
20.00	29.80	1002.20	3.02	30.50	956.60	3.04	30.10	1029.30	3.02	30.13	996.03	3.03	6.80
30.00	36.50	932.20	3.83	37.90	893.30	3.86	39.00	952.50	3.91	37.80	926.00	3.87	10.20
40.00	46.70	855.40	4.71	47.90	805.50	4.73	47.60	881.60	4.71	47.40	847.50	4.72	13.60
50.00	61.10	782.20	6.21	62.00	749.40	6.23	62.10	832.10	6.23	61.73	787.90	6.22	17.00
60.00	73.30	712.00	6.99	71.10	671.10	7.01	72.90	722.20	7.05	72.43	701.77	7.02	20.40
70.00	88.20	665.50	8.77	90.00	614.30	9.02	89.90	669.90	9.01	89.37	649.90	8.93	23.80
80.00	97.60	592.00	9.89	98.80	594.90	9.95	99.80	601.10	10.10	98.73	596.00	9.98	27.20

e) 150 minutes

RPM	Trial 1			Trial 2			Trial 3			Average			**S Rate (1/sec)**
	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	Torque (%)	**Viscosity (mPa.s)**	S Str (N/m2)	
0.00	0.10	0.00	0.01	0.20	0.00	0.01	0.20	0.00	0.01	0.17	0.00	0.01	0.00
1.00	1.87	1237.43	0.21	1.72	1240.32	0.20	2.13	1251.23	0.22	1.91	1242.99	0.21	0.34
10.00	18.70	1132.10	2.09	17.20	1053.40	2.03	21.30	1119.90	2.18	19.07	1101.80	2.10	3.40
20.00	29.80	1032.20	2.98	28.30	959.90	2.93	30.50	1012.20	3.04	29.53	1001.43	2.98	6.80
30.00	38.50	955.50	3.89	36.50	891.90	3.85	37.90	939.90	3.86	37.63	929.10	3.87	10.20
40.00	45.50	885.50	4.56	44.10	809.90	4.52	47.90	861.10	4.73	45.83	852.17	4.60	13.60
50.00	59.90	835.50	6.01	57.50	752.20	5.95	62.00	783.10	6.23	59.80	790.27	6.06	17.00
60.00	71.20	729.30	7.02	69.90	678.40	6.99	71.10	712.90	7.01	70.73	706.87	7.01	20.40
70.00	88.30	672.20	8.96	86.50	619.30	8.91	90.00	667.40	9.02	88.27	652.97	8.96	23.80
80.00	96.70	605.50	9.96	95.50	603.30	9.93	98.80	596.60	9.95	97.00	601.80	9.95	27.20