PRODUCTION & CHARACTERIZATION OF MULTI-WALLED CARBON NANOTUBE (MWCNT) REINFORCED POLYESTER

JOSHUA SINGH A/L MANTAR SINGH

BACHELOR OF CHEMICAL ENGINEERING (HONS)
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JOSHUA SINGH A/L MANTAR SINGH

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
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UNIVERSITI MALAYSIA PAHANG

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Signature	: ……
Name of main supervisor	: DR. MOHAMMAD DALOUR HOSSEN BEG
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Dedication

Special dedication to my supervisor, my family members, my friends, my fellow colleague and all faculty members for all your care, support and believe in me.

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ABSTRACT

This paper presents the study on the production and characterization of Hydroxyl functional 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 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 0.6wt % MWCNT. Hence, the optimum weight percentage of MWCNT is at 0.6wt %. After optimizing the weight percentage of MWCNT, the sonication time was optimized. This was done by preparing samples of 0.6wt % MWCNT which was then sonicated at different time intervals. After sonication, the viscosity was determined. From the result, it can be concluded that the optimum sonication time of 0.6wt % MWCNT is 1.5 hour. After optimization, the composite was then characterized and tested 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 0.6wt % MWCNT was stronger than the sample with pure Polyester. On the other hand, TGA results show that the optimized composite has higher thermal stability than pure Polyester. As a conclusion, this Polyester composite has the potential to be used in various applications due to its high mechanical strength and high thermal stability.

Keywords: *Nano-composite; Multi-Walled Carbon Nanotube (MWCNT); Polyester Resin, Viscosity, Sonication, Dispersion*

ABSTRAK

Kertas kerja ini membentangkan kajian mengenai pengeluaran dan pencirian komposit Poliester yang diperkukuhkan dengan Tiub Nano Karbon “Multi Walled” fungsi Hidroksil (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 0.6wt % MWCNT. Oleh itu, peratusan berat optimum MWCNT pada 0.6wt % MWCNT. Selepas mengoptimumkan peratusan berat MWCNT, masa sonikasi oleh komposit telah dioptimumkan. Ini dilakukan dengan menyediakan sampel 0.6wt % MWCNT yang kemudiannya telah disonikasi pada jangka masa yang berbeza. Selepas sonikasi, kelikatan ditentukan. Dari keputusan itu, ia boleh membuat kesimpulan bahawa masa sonikasi optimum 0.6wt % MWCNT adalah 1.5 jam. Selepas pengoptimuman, komposit itu kemudian dicirikan dan diuji 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 0.6wt % MWCNT adalah lebih kuat daripada sampel Polyester tulen. Oleh itu, masa sonikasi optimum untuk 0.6wt % MWCNT adalah 1.5 jam. 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 0.6wt % MWCNT lebih kuat daripada sampel tanpa MWCNT. Sebaliknya, keputusan TGA menunjukkan bahawa komposit yang sudah dioptimumkan itu mempunyai kestabilan terma yang lebih tinggi daripada sampel Polyester tulen. Kesimpulannya, poliester komposit ini mempunyai potensi untuk digunakan dalam pelbagai aplikasi kerana kekuatan yang tinggi mekanikal dan kestabilan terma yang tinggi.

Kata kunci: *Nano-komposit; Tiub Nano Karbon “Multi-Walled”; Resin Poliester; Kelikatan; Sonikasi: Serakan*

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LIST OF ABBREVIATIONS

CNTs	Carbon Nanotubes
CNT	Carbon Nanotube
MWCNTs	Multi-Walled Carbon Nanotubes
MWCNT	Multi-Walled Carbon Nanotube
DWCNTs	Double-Walled Carbon Nanotubes
DWCNT	Double-Walled Carbon Nanotube
SWCNTs	Single-Walled Carbon Nanotubes
SWCNT	Single-Walled Carbon Nanotube
CB	Carbon Black
THF	Tetrahydrofuran
MEKP	Methyl Ethyl Ketone Peroxide
TGA	Thermal Gravimetric Analysis
FTIR	Fourier Transform Infrared Spectroscopy
TS	Tensile Strength
wt %	Weight Percentage

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. By definition, a composite, in the present context, is a multiphase material that is artificially made, as opposed to one that occurs or forms naturally (Callister & Rethwisch, 2008).

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 (Callister & Rethwisch, 2008).

In composites, there are two main phases which is commonly known as the matrix phase and the reinforcement phase. The matrix phase is a continuous phase which binds the reinforcement in place whereas the reinforcement phase is the dispersed phase which improves the properties of the overall composite (Callister & Rethwisch, 2008). In this research, the matrix phase chosen was Polyester Resin whereas the reinforcement phase chosen was Hydroxyl-functional Multi-Walled Carbon Nanotubes (MWCNTs).

1.2 Motivation

The motivation for this study was largely initiated by the unique properties of Carbon Nanotubes (CNTs). From the literature, we know that CNT possess many unique characteristics and extraordinary specific properties such as high elastic modulus, tensile strength, thermal and electrical conductivity (Aurilia et al., 2012). In addition, CNT has also other excellent properties such as high flexibility, low mass density and large aspect ratio (typically>1000) (Spitalsky et al., 2010). Hence, through the incorporation of CNTs in a composite, the properties of the overall composite could be improved largely.

Moreover, the addition of CNTs into polymeric materials can be a great finding to the world of research. According to Aurilia et al. (2012), the addition of CNTs into polymeric materials will be a valuable solution in performances enhancement. However, this will only occur when CNTs are adequately dispersed. Nevertheless, in this research, the incorporation of MWCNTs in Polyesters was studied for potential applications in near future.

1.3 Problem Statement

There are two main challenges for developing high performance CNT/polymer nano-composites. The first challenge is to obtain a good homogeneous dispersion of CNTs in the polymeric matrix (Liu et al., 2004). This is because CNTs tends to bundle together and form some agglomeration due to intrinsic van der Waals attraction between the individual tubes (Dresselhaus et al., 2001). Hence, the agglomeration of CNTs in the polymer matrix will further yield a material with very low dispersion.

On the other hand, the second challenge is to obtain a good interfacial adhesion between CNT and polymer matrix (Liu et al., 2004). A weak interfacial bond between the nanotubes and the polymer matrix will limit the efficient load transfer to the polymer matrix and further cause a limitation in CNTs reinforcement role in the polymeric matrix (Lourie et al., 1998). Hence, the interfacial interaction between the CNTs and the polymer matrix should be strong enough for the efficient load transfer of polymeric matrix to the CNTs (Ma et al., 2010).

Hence, in this research, both drawbacks of the MWCNT/Polyesters system were overcome by improving the dispersion of MWCNTs in the system. Firstly, the weight percentage of MWCNT in the composite (when no solvent was used) was studied and optimized. Secondly, the weight percentage of MWCNT in the composite (when solvent was used) was studied and optimized. Thirdly, the sonication time needed by the composite for adequate dispersion would also be studied and optimized. After optimization, the produced composite was characterized and tested using Tensile Strength (TS) test, Thermal Gravimetric Analysis (TGA) and Fourier Transform Infrared Spectroscopy (FTIR).

1.4 Objectives

There are two main objectives in this study.

a) To produce MWCNTs reinforced Polyester
b) To characterize MWCNTs reinforced Polyester

1.5 Scope

This study will be primarily focused on the production of MWCNT reinforced Polyester and the characterization of the MWCNT reinforced Polyester. The area of the study narrows to the following:

a) To study and optimize the weight percentage of MWCNT in the composite when no solvent was used.
b) To study and optimize the weight percentage of MWCNT in the composite when solvent was used
c) To compare the optimization of MWCNTs weight percentage in the composite for both with and without solvent.
d) To study and optimize the sonication time needed by the composite for adequate dispersion.
e) To characterize, test and compare the MWCNT reinforced Polyester composite and Pure Polyester sample using various analyses such as Tensile Strength (TS) test, Thermal Gravimetric Analysis (TGA), and Fourier Transform Infrared Spectroscopy (FTIR).

1.6 Organisation of this Thesis

In this section, the structure of this thesis was outlined. Chapter 2 provides the literature review on composites, polymer nano-composites, Unsaturated Polyester (UP) Resin, Carbon Nanotubes (CNTs), dispersion of Carbon Nanotubes (CNTs), dispersion techniques and the properties of CNT/Polyester nano-composites.

Chapter 3 gives description on the materials, chemicals, glassware and apparatus used in this research. Moreover, the experiment procedure for each study was discussed in detail. Chapter 4 provides the results and discussions of this study. The result of the optimized weight percentage and sonication time is included. In addition, the result of Tensile Strength (TS) test, Thermal Gravimetric Analysis (TGA) and Fourier Transform

Infrared Spectroscopy (FTIR) is also included. Chapter 5 draws together a summary of the thesis, outlines the recommendation of this work and future work of this research. Finally, all the related raw data that was obtained in this study are included in the Appendix.

2 LITERATURE REVIEW

2.1 Overview

In this chapter, there will be literature review on composites, polymer nano-composites, Unsaturated Polyester (UP) Resin, Carbon Nanotubes (CNTs), dispersion of Carbon Nanotubes (CNTs), dispersion techniques and the properties of CNT/Polyester nano-composites.

2.2 Composites

In recent years, many of our modern technologies require a specific material which exhibits unusual combinations of properties which cannot be met by the conventional metal alloys, ceramics, and polymeric materials. This is true for materials that need to be utilized in fields such as aerospace, underwater and transportation applications. Through the development of composite materials, the research on the combination of materials property and ranges are being extended (Callister & Rethwisch, 2008).

As explained earlier in Chapter 1, there are two main phases in composites which is commonly known as the matrix phase and the reinforcement phase. The matrix phase is the continuous phase in the composite which binds the reinforcement in place whereas the reinforcement phase is actually the dispersed phase in the composite. The properties of composites are actually a function of the properties of both phases, their relative amounts, and the geometry of the dispersed phase (Callister & Rethwisch, 2008).

Finally, there are three types of classifications for composite materials. They are particle-reinforced, fiber-reinforced, and structural composites (Callister & Rethwisch, 2008). In this study, the composite material that was produced and characterized is actually particle-reinforced polymer nano-composite. In the next section, polymer nano-composites will be discussed even more thoroughly.

2.3 Polymer Nano-Composites

Polymer composites are composites that consist of additives and polymer matrices, which are made of thermoplastics, thermosets and elastomers. They are relatively inexpensive materials which can be utilized for various engineering applications. The

polymer composite is usually made up of two or more materials which possess unique properties that cannot be obtained when each material are acting alone (Ma et al., 2010).

The polymer composites are used for a great variety of applications. This is because they have many features such as low cost, low weight, corrosion resistance and also the ease of processing and shaping. However, the biggest drawback of the polymer composite is the mechanical properties of the composite. Hence, optimizing the mechanical properties has always been the most desired objective of researches (Al-Saleh & Sundararaj, 2011).

Traditionally, aromatic organic fibers such as Aramid and inorganic fibers such as glass and carbon fibers were used as fillers. It was initially used to boost the mechanical properties of polymers. However, more interest was developed in polymer nano-composites because nano-composites offers enhanced properties when compared to conventional polymer based composites which utilize micro-scale particulates such as Carbon Black (CB) or glass micro spheres (Gojny et al., 2004). Another reason for the development of polymer nano-composites is that when a material with multifunctional properties such as mechanical, electrical, thermal and etc. is required, carbon based nano-structured polymeric materials are normally favoured (Al-Saleh & Sundararaj, 2011). In addition, by using nano-composites, the properties of composites can be largely modified even at extremely low content of fillers. This is done by the incorporation of nano-scale CNTs into a polymer system which results in a very short distance between fillers (Ma et al., 2010).

Although nano-composite materials are increasingly commercially available, these materials are still very expensive. In order to make a successful nano-composite, it is very important to be able to disperse the filler material thoroughly throughout the matrix to maximize the interaction between the intermixed phases (Fink, 2013; Kim et al., 2009) and to have a good interfacial adhesion between CNT and polymer matrix (Kim et al., 2009). This is because nanotubes and other nano-fillers give rise to a very large fraction of interphase region in the composite (Reynaud et al., 1999).

2.4 CNT/Polymer Nano-Composites

Recently, CNT based polymeric materials have been the subject of investigation (Paul & Robeson, 2008). This is because CNT/polymer nano-composites has the potential to

be used as applications in various fields such as heat resistance, chemical sensing, electrical and thermal management, photoemission, electromagnetic absorbing and energy storage performances, and etc. (Ma et al., 2010).

CNT/polymer nano-composites can be classified in two major ways which is structural or functional composites (Du et al., 2007). As for structural composites, the mechanical properties of CNTs are explored to obtain structural materials with improved mechanical properties. As for the functional composites, other unique properties of CNTs such as electrical, thermal, optical and damping properties are utilized to develop multi-functional composites (Ma et al., 2010).

Previously, there have been many works on CNT/polymer systems such as Ma et al. (2003) whom synthesized CNT/polymer systems using Polyester. However, the biggest problem of using CNTs as reinforcement is that they have a strong tendency to agglomerate. This has an adverse effect on the properties of the composites because agglomeration reduces the amount of interphase region and creates stress concentration sites which initiate failure. In addition, it also reduces the load carrying capacity of the composite (Patel et al., 2008).

Hence, in this research, the dispersion of Carbon Nanotube (CNT) in the Polyester Resin will be studied and optimized. The dispersion of CNT will be improved by optimizing the weight percentage of CNT in Polyester Resin and the sonication time of the composite.

2.5 Unsaturated Polyester (UP) Resin

According to Ring et al. (2002), the global production/consumption of Unsaturated Polyester (UP) Resins in the year 2001 was 1.6 Million Metric Tons. The usage of UP is very high because UP Resins are commonly used as matrices for fiber-reinforced and glass fiber-reinforced composites (Kosar & Gomzi, 2010; Seyhan et al., 2007). Moreover, UP Resins are also thermosets (Fink, 2013), which have been utilized in many applications including automotive, construction, transportation, storage tanks and piping industry (Seyhan et al., 2007).

UP Resins consist of two types of polymers. The two types of polymers are short-chain polyester which contains double bonds and vinyl monomer. During the curing reaction,

the double bonds of the polyester will co-polymerize with the vinyl monomer. Hence, a three-dimensional network will be formed in the course of curing (Fink, 2013).

There are notable differences between Unsaturated Polyesters (UP) and Unsaturated Polyester (UP) Resins. Unsaturated Polyesters (UP) is the freshly synthesised polyesters that leave directly from the condensation vessel. They are rarely sold as such, because they are difficult to handle and are very brittle at room temperature. Instead, the freshly synthesised polyesters are mixed with the vinyl monomer in the molten state. This will further cause the material to be viscous at room temperature. This mixture of Unsaturated Polyester with the vinyl polymer is known as Unsaturated Polyester Resin and in this form it is commonly sold (Fink, 2013). The miscibility of the resin depends on the resin composition (Seyhan et al., 2007).

The advantage of using Polyester Resins is that it is versatile, quick curing, and has a long shelf life at room temperature (Seyhan et al., 2007). On the other hand, the biggest drawbacks of using UP Resin are that the double bonds in it are very easily affected by impurities or by the ambient temperature at the stage of storing. When the ambient storage temperature is higher or the storage time is longer, the danger of preliminary self-polymerization is higher. When the UP Resin self-polymerizes, an undesired gelation of the resin occurs which makes the resin unusable (Fink, 2013). A monetary loss due to deterioration of the workability of the resin occurs (Huang et al., 2011). In addition, UP resins has significantly higher cure shrinkage, as compared to epoxy (Seyhan et al., 2007).

2.6 Carbon Nanotubes (CNTs)

The first discovery of Carbon Nanotubes (CNTs) was by Iijima in the year 1991 (Iijima, 1991). Since then, CNT has attracted many researches to do study for potential applications in wide range of industry (Dumee et al., 2013). However, due to limited availability and higher cost, there are only a few practical applications in industrial field (Ma et al., 2003).

Carbon Nanotubes (CNTs) have diverse arrangements at a nano-metric level that lead to different properties depending on the specific kind of nanotubes. A number of different types of nanotubes, from Single-Walled Carbon Nanotubes (SWCNT), Double-Walled Carbon Nanotubes (DWCNT), and Multi-Walled Carbon Nanotubes (MWCNT) to their

variants with helix and bamboo shapes are already known to this date (Velasco-Santos et al., 2005). SWCNTs consist of a single graphene layer rolled up into a seamless cylinder whereas MWCNTs consist of two or more concentric cylindrical shells of graphene sheets coaxially arranged around a central hollow core with van der Waals forces between adjacent layers (Ma et al., 2010).

CNTs are different from other carbon materials, such as graphite, diamond and fullerene (C_{60}, C_{70}, etc.). This is because CNTs are one-dimensional carbon materials which can have an aspect ratio greater than 1000 (Ma et al., 2010). The aspect ratio of CNT is very high because CNTs are composed of thin tubes with diameters of only a few nano-meters, but a length of few microns (Seyhan et al., 2007). The cylindrical nanotube usually has at least one end capped with a hemisphere of fullerene structure (Iijima, 1991). This special property gives CNT to exhibit higher aspect ratio, extraordinary mechanical, thermal and electrical properties, which make them prime candidates as reinforcing constituents in various polymers for the production of nano-composites (Seyhan et al., 2007; Esawi & Farag, 2007). Although each single CNT has a very high aspect ratio (length to diameter ratio), and consequently very high specific surface area (S_v), CNT tends to agglomerate. This is due to non-homogeneous dispersion and random distribution of the nanotubes inside the resin (Seyhan et al., 2007).

CNTs have three types of chirality which is known as armchair, zigzag and chiral. The tube chirality is defined by the chiral vector, Ch. The formula is as follows:

$$C_h = na_1 + ma_2$$

where the integers (n,m) are the number of steps along the unit vectors (a_1 and a_2) of the hexagonal lattice (Dresselhaus et al., 1995).

By using (n,m) naming scheme, the three types of orientation of the carbon atoms around the nanotube circumference can be specified. Firstly, if n = m, the nanotubes are called “armchair”. Secondly, if m = 0, the nanotubes are called “zigzag”. Thirdly, if they are otherwise, they will be called as “chiral”. The chirality of nanotubes is important because it has significant impact on its transport properties especially for the electronic properties. For a given (n,m) nanotube, if (2n + m) is a multiple of 3, then the nanotube is a metallic, otherwise the nanotube is a semiconductor. Each MWCNT contains a multi-layer of graphene, and each layer can have different chirality, so the

prediction of its physical properties is more complicated than that of SWCNT (Ma et al., 2010).

CNTs are the strongest and stiffest materials on earth due to its chemical bonding which is composed entirely of sp^2 carbon-carbon bonds. This bonding structure is relatively higher than the sp^3 bonds which are found in diamond. This bonding structure provides CNTs with extremely high mechanical properties (Ma et al., 2010). The outstanding mechanical properties exhibited by CNTs are high Young's modulus, stiffness and flexibility (Velasco-Santos et al., 2005). In addition, CNTs have unique electronic properties may suggest possibilities for use as either semiconductor or metallic conductive nano-materials (Ebbesen et al., 1996). Also, these structures possess high thermal stability (Che et al., 2000) which could be advantageous for aerospace applications (Velasco-Santos et al., 2005).

Agnihotri et al. (2011) found out that even though CNTs have excellent mechanical properties, their incorporation in polymer matrices do not necessarily result in dramatically improved composites. In addition, Shu-quan et al. (2007) found out that initially tensile strength and modulus increase with increasing filler loading of CNTs. Beyond a critical mass fraction, the CNTs have a deteriorating effect on the strength and modulus of the composite. On the other hand, Wong et al. (2003) have found that increasing the weight fraction of Multi-Walled Carbon Nanotubes (MWCNTs) beyond a critical value has an adverse effect on failure strain, tensile strength and elastic modulus of MWCNT/polymeric composites. Clearly, an optimal loading of CNTs in the polymer matrix is a key parameter to harness its excellent mechanical properties in structural composites.

Mechanical properties of Carbon Nanotube (CNT) reinforced polymer composites are not entirely established due to the fact that CNT have a very strong tendency to exist in agglomerated form. Therefore, homogeneous dispersion of CNT in the polymer matrix is one of the key factors to enhance mechanical properties of the composites (Seyhan et al., 2007; Geng et al., 2002).

2.7 Dispersion of Carbon Nanotube (CNT)

The degree of dispersion is commonly regarded as functions of the size of the dispersed particles, wettability by disperse medium and nature of the attractive forces between the corresponding constituents (Kinloch et al., 2002).

According to Liu & Choi (2012), dispersion is a spatial property whereby the individual CNT are spread with the roughly uniform number density throughout the polymer matrix which is in continuous phase. The first challenge is to separate the tubes from their initial aggregated assemblies. This is normally achieved by local shear force such as direct manual mixing of CNTs with polymer resin. However, this does not create sufficient local shear force and therefore lead to a poor dispersion of CNTs inside the polymer matrix. The more effective separation of CNT bundles requires the overcoming of the inter-tube Van der Walls forces of attraction.

The dispersion of CNT in polymer matrix could be assisted by the introduction of a common solvent which is able to dissolve polymer resin easily and at the same time allows dispersion of CNT. In this case, two dispensed solutions sharing common solvent but containing polymer resin and CNT respectively. After that, the solution undergoes mechanical stirring and sonication process. Finally, with the complete evaporation of solvent, CNTs would leave dispersed in the polymer (Liu & Choi, 2012).

The choice of organic solvent is critical to determine the final dispersion quality and it also depends on the polymer matrix. Although common solvent is used to help dispersion, the optimization of process conditions is still critical to ensure final dispersion quality (Liu & Choi, 2012). The common utilized solvents are Tetrahydrofuran (Chua et al., 2012) and Chloroform (Kohlmeyer et al., 2009).

2.8 Dispersion Techniques

2.8.1 Introduction

There are two different methods to disperse nanotubes which are mechanical or physical methods and chemical methods. Mechanical dispersion methods such as ultrasonication, separate nanotubes from each other, but can also fragment it and decrease its aspect ratio while processing. On the other hand, chemical methods that uses surfactants or functionalization to change their surface energy, which further improves its wetting or

adhesion characteristic and also reduces 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 (Hilding et al., 2003).

The common dispersion techniques for processing CNT/polymer composites have been direct mixing, ultrasonication (Thostenson et al., 2005; Seyhan et al., 2007; Gojny et al., 2005), calendering (Shokrieh et al., 2014) and high pressure homogenizing (Jia et al., 2012). In addition, Gojny et al. (2005) has also used the 3-roll milling to disperse CNT in an epoxy resin. They further concluded that 3-roll milling technique provided a better dispersion of CNTs in the epoxy resin and also result in higher mechanical properties, as compared to those prepared by sonication. Besides the physical approaches for the CNT dispersion, there are also some other attempts including the use of surfactants and chemical functionalization of the CNT-surfaces that had been made to alter the degree of dispersion and to tailor the interface between the matrix and the CNT (Seyhan et al., 2007).

2.8.2 Ultrasonication

Ultrasonication is a method whereby ultrasound or ultrasonic energy will be applied to a solution in order to agitate the particles for various purposes. In laboratory, it is usually achieved using an Ultrasound or Ultrasonic Water Bath which is also known as sonicator (Ma et al., 2010).

Ultrasonication is the most frequently used method for nano-particle dispersion. When ultrasound propagates by a series of compression, waves are induced in the molecules of the medium through in which it passes. The production of these shock waves promotes the “peeling off” of individual nanoparticles which are located at the outer part of the nanoparticle bundles, or agglomerates, and thus results in the separation of individualized nanoparticles from the bundles (Ma et al., 2010).

Generally, ultrasonication is an effective method to disperse CNTs in liquids which have a low viscosity, such as water, acetone and ethanol. However, to sonicate polymers which exist as a viscous liquid, the polymer needs to be dissolved or diluted using any solvent. The probe of the Ultrasonic Water bath is usually made of inert material such

as titanium. They are attached with a base unit and tapered down to a tip with a certain diameter. This is important so that the energy from the wide base is focused on the tip. This gives the probe a high intensity. However, the biggest consequence of this configuration is that during sonication, it can generate substantial heat rapidly. Therefore, when volatile solvents such as ethanol and acetone are used, the samples should be kept cold by using an ice bath and the sonication must be done in short intervals (Ma et al., 2010).

Seyhan et al. (2007) applied sonication method with the CNT/resin systems. They concluded that although the sonication bath was cooled by water, the local heating due to energy created within the resin system, caused styrene evaporation from the polymer suspension, leading to a more viscous resin. They also noticed that the CNTs that were closer to the tip of the sonicator were agglomerated. This is because of the Van der Wall's attractive force between the CNT-surfaces which are known to be sensitive to heat that further cause's agglomeration (Thostenson et al., 2005). Seyhan et al., (2007) also discovered that Multi-Walled CNTs with functional groups exhibited better local dispersion in the polyester matrix, as compared to Double-Walled CNTs with and without treatment. In general, Double-Walled CNTs were observed to be more agglomerated form caused by their pronounced higher surface area.

2.9 Properties of CNT/Polyester Nano-Composites

2.9.1 Curing

Curing is achieved in general with a radical initiator. Even when a wide variety of initiators are available, common peroxides used are ketone peroxides which includes Methyl Ethyl Ketone Peroxide (MEKP) and Acetyl Acetone Peroxide (Fink, 2013).

Seyhan et al. (2007) used Methyl Ethyl Ketone Peroxide (MEKP) as an initiator to polymerize the resin suspensions that contain various amounts of CNTs. MEKP is known as organic peroxide which is formed by the reaction of Methyl Ethyl Ketone (MEK) and with Hydrogen Peroxide. Even in industry, MEKP is widely used as an initiator or cross-polymerisation agent in polymer fabrication processes. Undiluted MEKP possesses explosive properties and, as such, commercial supply is as diluted formulations containing a substance such as dimethyl phthalate (DMP) (Graham et al., 2011).

2.9.2 Adhesion

Adhesion between filler and polymer matrix could by physical, chemical and/or mechanical. Other forms of adhesion are diffusive and electrostatic but these are not common in polymer composites (Al-Saleh & Sundararaj, 2011).

Physical adhesion refers to the intermolecular forces, such as van der Waals forces, between filler and polymer matrix, which is the most common type of adhesion in polymer composites. Chemical adhesion represents chemical bonding, such as covalent bond, between the matrix and filler, which is typically the strongest form of adhesion. Filler particles are typically functionalized with certain chemical group in order to achieve good bonding with the polymer matrix. Mechanical adhesion represents the interlocking and entanglement of polymer chains within the filler structural voids and entanglement between filler functional chains and polymer matrix (Al-Saleh & Sundararaj, 2011).

2.10 Summary

The paper presents on the production and characterization of Multi-Walled Carbon Nanotubes (MWCNTs) reinforced Polyester. In this chapter, the reviews from previous work have been cited. In the next chapter, there will be description on the materials, chemicals, glassware and apparatus used in this research. Moreover, the experiment procedures for each study will be explained in detail.

3 MATERIALS AND METHODS

3.1 Overview

In this chapter, the methodology of producing MWCNT reinforced Polyester will be discussed in detail. There are 4 main parts in this experiment. Firstly, the optimum weight percentage of MWCNT in the composite when no solvent was used will be determined. Secondly, the optimum weight percentage of MWCNT in the composite when THF was used as solvent will be determined. Thirdly, the optimum sonication time needed by the composite for adequate dispersion will be determined. Finally, the optimized MWCNT reinforced Polyester composite will be characterized, tested and compared using various methods such as Tensile Strength (TS) test, Thermal Gravimetric Analysis (TGA) and Fourier Transform Infrared Spectroscopy (FTIR).

3.2 Chemicals

3.2.1 Polyester Resin

The primary material which was chosen as the matrix and continuous phase was Polyester Resin. Polyester Resin is a pink colour liquid resin which is also known as Bicarbonate Haemodialysis Concentrate BP Acidic Component (Solution A). The polyester resin was manufactured by Ain Medicare Sdn Bhd and was provided by my supervisor, Dr. Mohammad Dalour Hossen Beg.

Figure 3.1: Polyester Resin

3.2.2 Multi-Walled Carbon Nanotube (MWCNT)

The primary material that was chosen as the reinforcement phase for this composite is known as Hydroxyl functional Multi-Walled Carbon Nanotube (MWCNT). MWCNT is a black coloured powder which was manufactured in China. MWCNT was provided by my supervisor, Dr. Mohammad Dalour Hossen Beg.

Figure 3.2: Hydroxyl functional MWCNT

3.2.3 Tetrahydrofuran (THF)

In this study, Tetrahydrofuran was used as a solvent. THF was manufactured by R&M Chemicals and was purchased from Impian Z Enterprise.

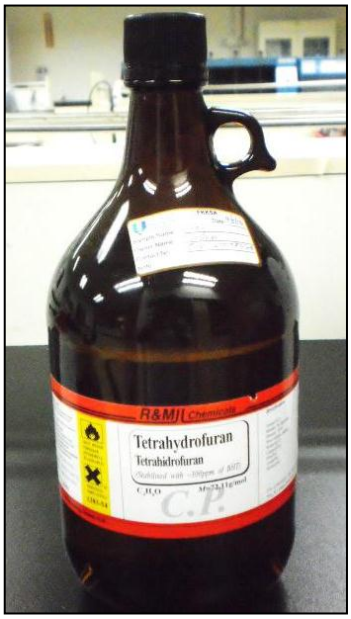

Figure 3.3: Tetrahydrofuran (THF)

3.2.4 Acetone

Acetone was used to clean the glassware and apparatus used in this study. Acetone was provided by Universiti Malaysia Pahang (UMP) FKKSA Laboratory.

Figure 3.4: Acetone

3.2.5 Methyl Ethyl Ketone Peroxide (MEKP)

In this study, Methyl Ethyl Ketone Peroxide (MEKP) was used as an initiator to initiate the crosslinking of the Polyester Resin. MEKP was manufactured by Sigma-Aldrich and was purchased from Impian Z Enterprise.

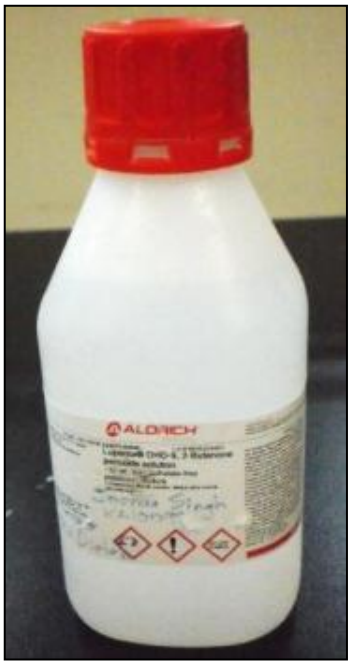

Figure 3.5: Methyl Ethyl Ketone Peroxide (MEKP)

3.3 Glassware

The glassware used in this study were beakers, measuring cylinders, glass rod, vials, spatula, thermometer, petri dish, magnetic stirrer, and etc. All the needed glassware for this study was obtained from Universiti Malaysia Pahang (UMP) FKKSA Laboratory.

3.4 Apparatus

3.4.1 Weighing Balance

Weighing Balance was used to measure the weight of MWCNT. The weighing balance used in this study is located at the Environmental Engineering Laboratory in Universiti Malaysia Pahang (UMP) FKKSA Laboratory.

Figure 3.6: Weighing Balance

3.4.2 Oven

Oven was used to remove the moisture content from MWCNT powder. The oven used in this study is located at the Environmental Engineering Laboratory in Universiti Malaysia Pahang (UMP) FKKSA Laboratory. The oven was set at 100-105°C.

Figure 3.7: Oven

3.4.3 Stirring Hotplate

The Stirring Hotplate was used for direct mixing of MWCNT and Polyester Resin. The Stirring Hotplate used in this study is located at the Environmental Engineering Laboratory in Universiti Malaysia Pahang (UMP) FKKSA Laboratory.

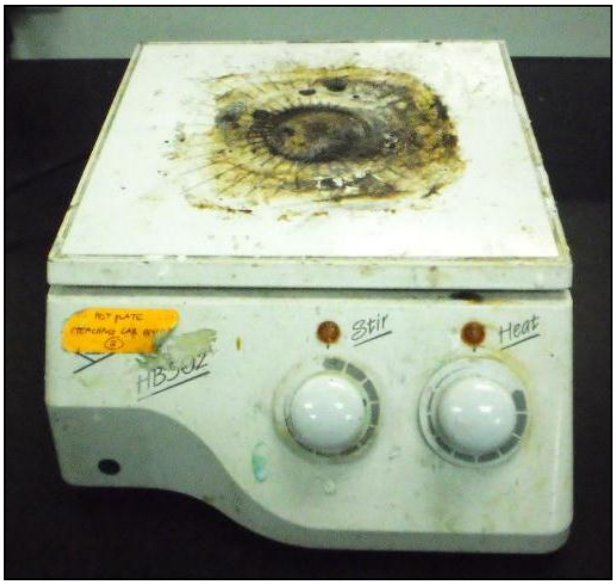

Figure 3.8: Stirring Hotplate

3.4.4 Ultrasonic Water Bath

Ultrasonic Water Bath was used to sonicate the MWCNT and Polyester composite. This is important so that the MWCNT are adequately dispersed in the Polyester Resin. The Ultrasonic Water Bath used in this study is located in Universiti Malaysia Pahang (UMP) FKKSA Laboratory.

Figure 3.9: Ultrasonic Water Bath

3.4.5 Metal Mold

Metal mold were used to shape the composite to the desired shape. The metal mold used in this study is located at the Environmental Engineering Laboratory in Universiti Malaysia Pahang (UMP) FKKSA Laboratory.

Figure 3.10: Metal Mold

3.4.6 Fume Hood

The entire experiment of this study was conducted inside the Fume Hood. This is because the chemicals used in this study are highly volatile and carcinogenic. The Fume Hood used in this study is located at the Environmental Engineering Laboratory in Universiti Malaysia Pahang (UMP) FKKSA Laboratory.

Figure 3.11: Fume Hood

3.4.7 Viscometer

Viscometer was used to determine the shear rate and the viscosity of the composite. The Viscometer used in this study is located in the Universiti Malaysia Pahang (UMP) FKKSA Laboratory.

Figure 3.12: Viscometer

3.4.8 Thermal Gravimetric Analysis (TGA)

TGA was used to determine the changes in the weight of the composite as a function of the temperature. TGA used in this study is located in the Universiti Malaysia Pahang (UMP) FKKSA Laboratory.

Figure 3.13: Thermal Gravimetric Analysis (TGA)

3.4.9 Fourier Transform Infrared Electroscopy (FTIR)

FTIR was used to obtain the spectral data of the composite. FTIR used in this study is located in the Universiti Malaysia Pahang (UMP) FKKSA Laboratory.

Figure 3.14: Fourier Transform Infrared Electroscopy (FTIR)

3.4.10 Universal Tensile Machine

UTM was used to test the tensile strength of the composite. UTM used in this study is located in the Universiti Malaysia Pahang FKKSA Laboratory.

Figure 3.15: Universal Tensile Machine (UTM)

3.5 Experimental

3.5.1 To Optimize the Weight Percentage of MWCNT in the Composite when No Solvent was used

The methods to determine the optimum weight percentage of MWCNT in the composite when no solvent was used will be discussed thoroughly in this section.

1) MWCNT powder was dried for one hour in a preheated oven at 100-105°C to remove any moisture content.
2) A sample with a total volume of 50mL was prepared. The sample was prepared by using a weight percentage of 0.1% MWCNT and 99.9% of Polyester Resin.
3) Both the measured MWCNT 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 Stirring Hotplate at full speed for about 10 minutes. No heating was required at this stage.
5) Then, the beaker was placed inside an Ultrasonic Water Bath for one hour. No heating was required at this stage.
6) After 1 hour of sonication, the composite was tested to determine its viscosity using Viscometer.
7) The procedure from 2-6 was repeated for different percentage of MWCNT which was 0.2%, 0.3%, 0.4%, 0.5%, 0.6%, 0.7% and 0.8%.
8) The results were recorded in the table below (The results are tabulated in Appendix A).

Table 3.1: Viscometer Results to Determine Optimum Weight Percentage of MWCNT in the composite when No Solvent was used

Trial	MWCNT (wt %)	MWCNT (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				
6	0.6	0.30				
7	0.7	0.35				
8	0.8	0.40				

3.5.2 To Optimize the Weight Percentage of MWCNT in the Composite when Solvent was used

The methods to determine the optimum weight percentage of MWCNT in the composite when solvent was used will be discussed thoroughly in this section.

1) MWCNT powder was dried for one hour in a preheated oven at 100-105°C to remove any moisture content.
2) A sample with a total volume of 50mL was prepared. The sample was prepared by using a weight percentage of 0.1% MWCNT and 99.9% of Polyester Resin.
3) Both the measured MWCNT and Polyester Resin were added into a 100mL beaker. In addition, Tetrahydrofuran (THF) was added into the beaker (Ratio of MWCNT to THF is 1:25). The mixture was stirred using glass rod for about 1-2 minutes.
4) After that, the mixture was stirred using Stirring Hotplate at full speed for about 10 minutes. No heating was required at this stage.
5) Then, the beaker was placed in an Ultrasonic Water Bath for 1 hour. No heating was required at this stage.
6) After 1 hour of sonication, the composite was tested to determine its viscosity using Viscometer.
7) The procedure from 2-6 was repeated for different percentage of MWCNT which was 0.2%, 0.3%, 0.4%, 0.5%, 0.6%, 0.7% and 0.8%.
8) The results were recorded in the table below (The results are tabulated in Appendix B).

Table 3.2: Viscometer Results to Determine Optimum Weight Percentage of MWCNT in the Composite when Using Tetrahydrofuran (THF) as Solvent

Trial	MWCNT (wt %)	MWCNT (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				
6	0.6	0.30				
7	0.7	0.35				
8	0.8	0.40				

3.5.3 To Determine the Optimum Sonication Time

The methods to determine the optimum sonication time needed by the composite for adequate dispersion will be discussed thoroughly in this section.

1) MWCNT powder was dried for one hour in a preheated oven at 100-105°C to remove any moisture content.
2) A sample with a total volume of 50mL was prepared. The sample was prepared by using a weight percentage of 0.1% MWCNT and 99.9% of Polyester Resin.
3) Both the measured MWCNT and Polyester Resin were added into a 100mL beaker. In addition, Tetrahydrofuran (THF) was added into the beaker (Ratio of MWCNT to THF is 1:25). The mixture was stirred using glass rod for about 1-2 minutes.
4) After that, the mixture was stirred using Stirring Hotplate at full speed for about 10 minutes. No heating was required at this stage.
5) Then, the beaker was placed in an Ultrasonic Water Bath for 30 minutes. No heating was required at this stage.
6) After 30 minutes of sonication, the solution was tested to determine its viscosity using Viscometer.
7) The procedure from 2-6 was repeated by using different sonication time which was 1 hour, 1.5 hour, 2 hour and 2.5 hour.
8) The results were recorded in the table below (The results are tabulated in Appendix C).

Table 3.3: Viscometer Results to Determine Optimum Sonication Time

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				

3.6 Characterization

3.6.1 Introduction

In this section, the methods to prepare the samples for characterization will be discussed. For characterization, two types of samples was produced which is Pure Polyester and the optimized composite (Polyester + 0.6wt % MWCNT). The various characterizations and testing include Tensile Strength (TS) test, TGA and FTIR.

3.6.2 Preparation of Pure Polyester Sample

A sample with a total volume of 250mL of pure Polyester Resin was prepared. Methyl Ethyl Ketone Peroxide (MEKP) was added to the Polyester Resin (Ratio of MEKP to resin was 1:100). The solution was stirred for 1-2 minutes to remove any trapped air bubbles. The mixture was poured into the metal mold. The mold containing the mixture was left to dry in the fume hood for two days. After two days, the solid composite was removed from the metal mold. Finally, the metal mold was cleaned using acetone for next usage.

3.6.3 Preparation of Pure Polyester + MWCNT composite

MWCNT powder was dried for one hour in a preheated oven at 100-105°C to remove any moisture content. A sample with a total volume of 250mL was prepared. The sample was prepared by using a weight percentage of 0.6% MWCNT and 99.4% of Polyester Resin. Both the measured MWCNT and Polyester Resin were added into a 500mL beaker. In addition, Tetrahydrofuran (THF) was added into the beaker (Ratio of MWCNT to THF is 1:25). The mixture was stirred using glass rod for about 1-2 minutes. After that, the mixture was stirred using Stirring Hotplate at full speed for about 10 minutes. No heating was required at this stage. Then, the beaker was placed in an Ultrasonic Water Bath. No heating was required at this stage. After 1.5 hours of sonication, the beaker was removed from the Ultrasonic Water Bath. 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 to remove any trapped air bubbles. The mixture was poured into the metal mold. The mold containing the mixture was left to dry in the fume hood for two days. After two days, the solid composite was removed from the metal mold. Finally, the metal mold was cleaned using acetone for next usage.

The major preparation steps are shown in the figures below:

Figure 3.16: Dried MWCNT was measured

Figure 3.17: Prepared Sample

Figure 3.18: Sample was stirred using Hotplate Stirrer

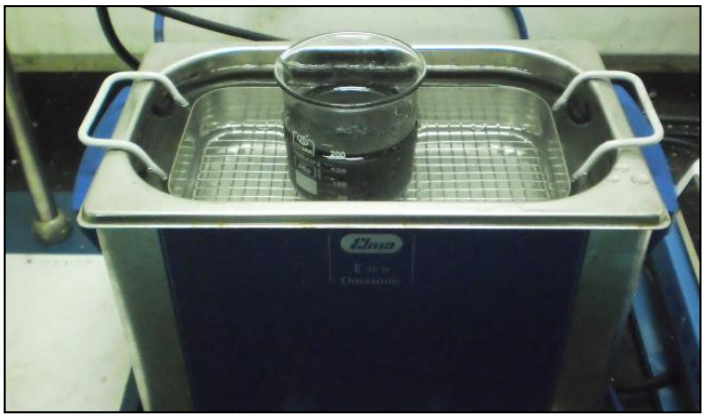

Figure 3.19: Sample was sonicated in Ultrasonic Water Bath

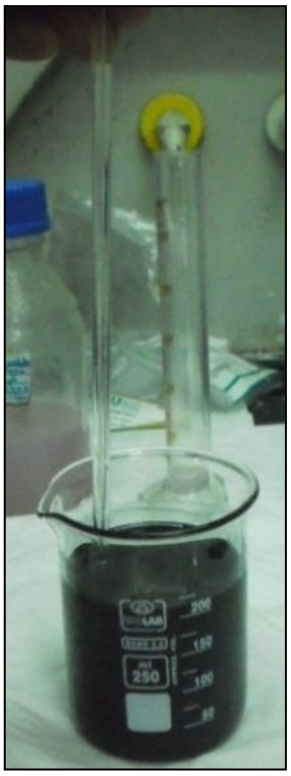

Figure 3.20: Sample was stirred after adding MEKP

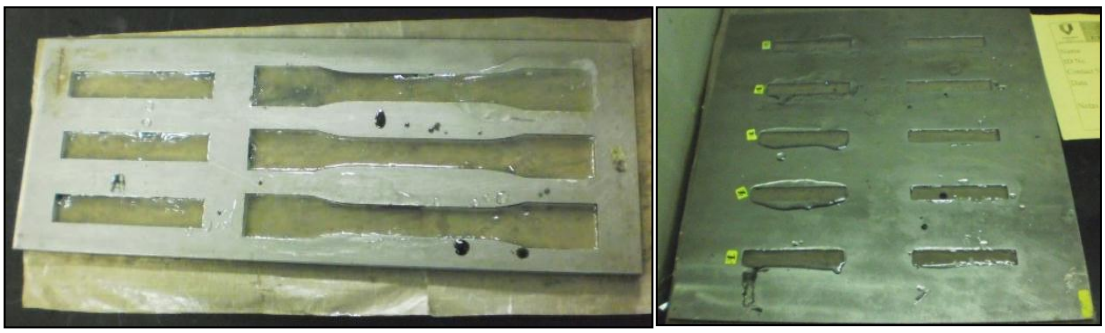

Figure 3.21: Pure Liquid Polyester Resin was poured into Metal Mold

Figure 3.22: Hardened Pure Polyester

Figure 3.23: Polyester Resin + 0.6wt % MWCNT was poured into Metal Mold

Figure 3.24: Hardened Composite (Polyester + 0.6wt % MWCNT)

4 RESULTS & DISCUSSION

4.1 Introduction

In this chapter, the results that were obtained from the experiment will be discussed. To improve the validity of the result, the average data of three samples were taken instead of one sample. Hence, all the figures shown in this chapter are representing average values. On the other hand, the raw data obtained for each sample are tabulated in Appendix. The results that was discussed in this chapter includes comparison of viscosity for different weight percentage of MWCNT when no solvent was used, comparison of viscosity for different weight percentage of MWCNT when solvent was used, comparison of viscosity for all weight percentage of MWCNT, comparison of sonication time, Tensile Strength (TS) test, Thermal Gravimetric Analysis (TGA), and Fourier Transform Infrared Spectroscopy (FTIR).

4.2 Optimization

4.2.1 Optimize Weight Percentage of MWCNT when No Solvent was used

Figure 4.1 shows the average viscosity versus shear rate for different weight percentage of MWCNT (0.1wt % to 0.8wt %) when no solvent was used. The raw data obtained for this experiment are tabulated in Appendix A.

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. Theoretically, a liquid that exhibits a property like this is known as a Non-Newtonian fluid (Jung et al., 2013). Nevertheless, from the result we can see that there is no viscosity reading beyond a certain shear rate. This is because when using the Viscometer, there will be no viscosity reading after the torque reaches 100%.

Secondly, the overall average viscosity increases when there is an increase in the weight percentage of MWCNT. For instance, at a shear rate of 3.40/sec, the average viscosity of MWCNT at 0.1wt % and 0.6wt % was 633.03mPa.s and 762.87mPa.s respectively.

However, from 0.6wt % MWCNT to 0.8wt % MWCNT, the average viscosity decreases from 762.87mPa.s to 676.23mPa.s respectively. This shows that the average viscosity is the highest at 0.6wt % MWCNT. As we know earlier, the higher the viscosity, the better the dispersion of MWCNT. Therefore, the optimum weight percentage or critical weight percentage of MWCNT is 0.6wt %.

Moreover, 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.

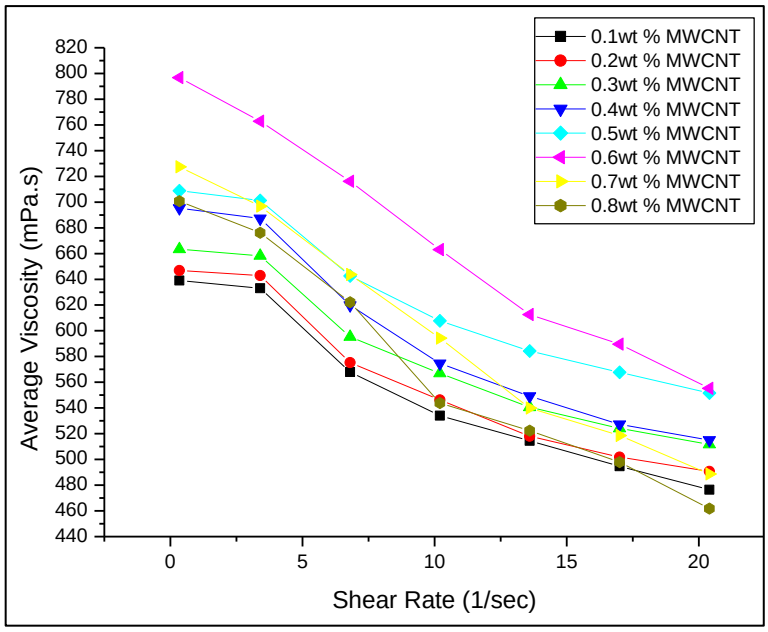

Figure 4.1: Average Viscosity versus Shear Rate for Different Weight Percentage of MWCNT (0.1wt % to 0.8wt %) when No Solvent was used

4.2.2 Optimize the Weight Percentage of MWCNT when Solvent was used

Figure 4.2 shows the average viscosity versus shear rate for different weight percentage of MWCNT (0.1wt % to 0.8wt %) when solvent was used. The raw data obtained for this experiment are tabulated in Appendix B.

Generally, the discussions that can be made from the graph are actually similar to the previous graph when no solvent was used. Firstly, the average viscosity of the

composite is also dependent on the shear rate. This proves that the composite is a Non-Newtonian fluid (Jung et al., 2013).

Secondly, the overall average viscosity increases when there is an increase in the weight percentage of MWCNT. For instance, at a shear rate of 3.40/sec, the average viscosity of MWCNT at 0.1wt % and 0.6wt % was 860.13mPa.s and 1043.63mPa.s respectively. However, from 0.6wt % MWCNT to 0.8wt % MWCNT, the average viscosity decreases from 1043.63mPa.s to 901.43mPa.s respectively. This shows that the average viscosity is the highest at 0.6wt % MWCNT which further leads to a better dispersion of MWCNT in Polyester Resin. Therefore, the optimum weight percentage or critical weight percentage of MWCNT is 0.6wt %.

Similarly, for this case, beyond the optimum weight percentage, the MWCNT has a deteriorating effect on the strength and modulus of the composite (Shu-quan et al., 2007). This is the reason for a lower average viscosity for 0.7wt % and 0.8wt %.

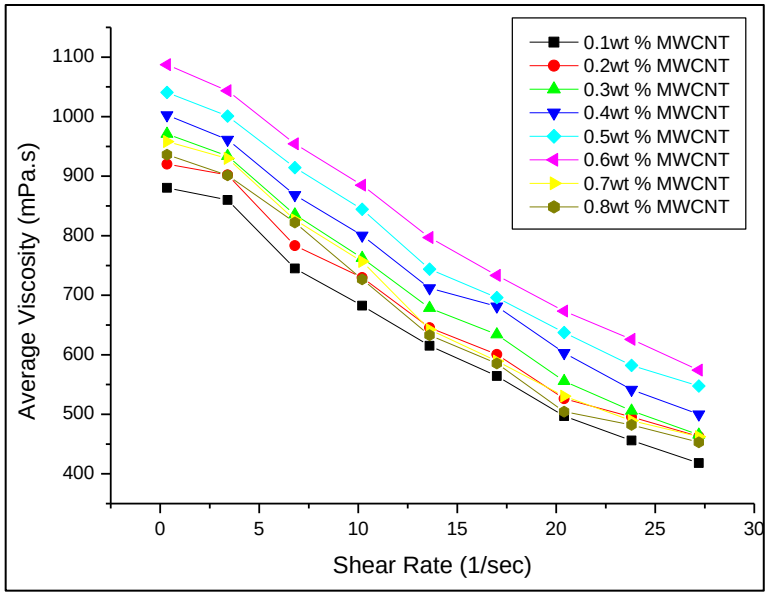

Figure 4.2: Average Viscosity versus Shear Rate for Different Weight Percentage of MWCNT (0.1wt % to 0.8wt %) when Solvent was used

4.2.3 Comparison of MWCNT's Weight Percentage for Both with & without Solvent

Figure 4.3 shows the comparison of average viscosity versus shear rate for different weight percentage of MWCNT (0.1wt % to 0.8wt %) when both without solvent and

with solvent were used. The raw data that was used to compare are tabulated in Appendix C.

From the graph, the overall average viscosity was relatively higher when solvent was used compared to the average viscosity when solvent was not used. For an example, the average viscosity at 0.6wt % MWCNT with solvent and without solvent was 1043.63mPa.s and 762.87mPa.s respectively. According to Liu & Choi (2012), the dispersion of MWCNT could be assisted by the introduction of a common solvent such as THF which dissolves polymer resin easily and at the same time allows mono-dispersion of MWCNT. This proves that the addition of a solvent can increase the average viscosity which further increases the dispersion of MWCNT in the composite.

Another important thing to note is that the average viscosity values of the optimized composite when solvent was not used is even lower than the average viscosity of 0.1wt % MWCNT when solvent was used. In other words, without using any solvent, the average viscosity data will be very low when compared to average viscosity when solvent was used.

As a conclusion, the optimum weight percentage of MWCNT in the composite is 0.6wt % with the use of solvent. In the following section, the dispersion of MWCNT in the composite will be further improved by optimizing the sonication time of the composite.

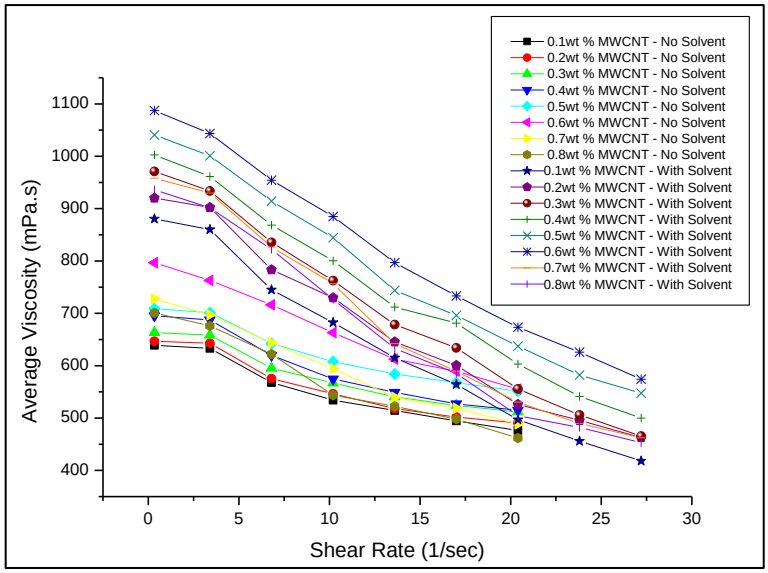

Figure 4.3: Comparison of Average Viscosity versus Shear Rate for Different Weight Percentage of MWCNT (0.1wt % to 0.8wt %)

4.2.4 Optimizing the Sonication Time

In this experiment, the dispersion of MWCNT in the composite was further improved by optimizing the sonication time of the composite. Figure 4.4 shows the average viscosity versus shear rate for different sonication time. The raw data obtained for this experiment are tabulated in Appendix D. The experiment was done by sonicating samples of 0.6wt % MWCNT at different time intervals.

There are some notable discussions that can be made from the figure below. Firstly, the average viscosity of the composite is dependent on the shear rate. Similarly, the composites can be validated as Non-Newtonian fluid (Jung et al., 2013).

Secondly, the average viscosity is very low after sonicating for 0.5 hour. 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 0.5 hour, the average viscosity increases drastically until 1.5 hour. Beyond 1.5 hours of sonication, there is very small increment in the average viscosity of the composite. This is because at 1.5 hour, the MWCNT has fully dispersed in the Polyester Resin. Therefore, any further sonication does not bring any notable effect on the overall dispersion of the composite.

However, the critical or optimum sonication time that was chosen was 1.5 hour. This is because from 1.5 hour to 2.5 hour, there is very small increment in the average viscosity. Waiting for a long time just to sonicate the composite for very small changes in dispersion or average viscosity is not practically feasible. Sadly, there are no literatures or study from previous work on optimizing the sonication time.

As a conclusion, the optimum sonication time of the composite is 1.5 hour whereas the optimum weight percentage of MWCNT is 0.6wt % with the usage of solvent. In the following section, the composite will be prepared based on the optimizations and will be characterized using Tensile Strength (TS) test, TGA and FTIR.

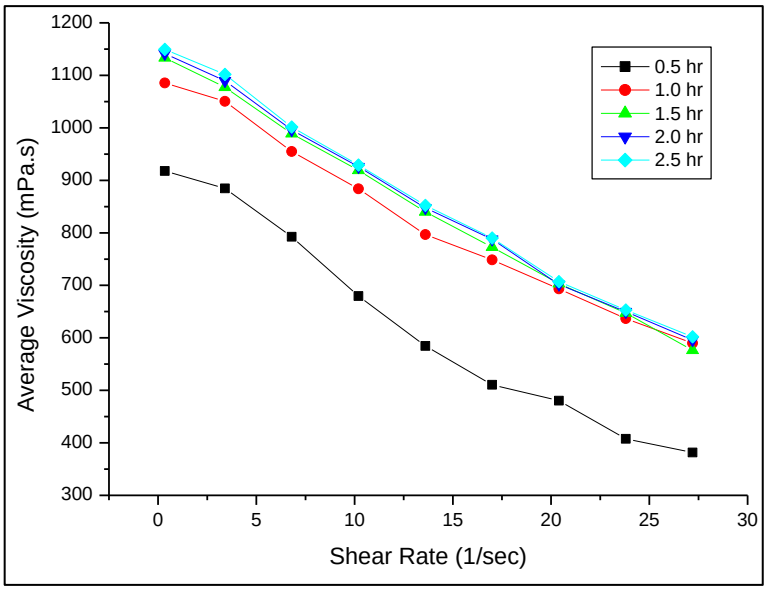

Figure 4.4: Average Viscosity versus Shear Rate for Different Sonication Time

4.3 Characterization

For characterization, two types of samples were tested and compared. The samples are pure Polyester sample and the optimized composite (Polyester + 0.6wt % MWCNT).

4.3.1 Tensile Strength (TS) Test

The samples were tested using Tensile Strength (TS) Test to determine the maximum force, maximum stress and maximum strain. The data obtained for pure Polyester and the optimized composite is tabulated in Table 4.1 and Table 4.2 respectively.

Table 4.1: Tensile Strength (TS) data for Pure Polyester sample

Name	Max Force (N)	Max Stress (N/mm^2)	Max Strain (%)
Pure Resin – Sample 1	959.04	19.98	2.90
Pure Resin – Sample 2	1087.97	22.67	3.01
Pure Resin – Sample 3	1480.98	30.85	4.63
Average	**1176.00**	**24.50**	**3.51**

Table 4.2: Tensile Strength (TS) data for optimized composite sample

Name	Max Force (N)	Max Stress (N/mm^2)	Max Strain (%)
0.6wt % MWCNT – Sample 1	1982.20	43.30	4.63
0.6wt % MWCNT – Sample 2	1898.20	38.90	3.54
0.6wt % MWCNT – Sample 3	1964.40	40.10	3.89
Average	**1948.27**	**40.77**	**4.02**

There are a number of discussions that can be made from the table above. Firstly, the average maximum force of pure Polyester and the optimized composite was 1176.00 N and 1948.27 N respectively. This shows that the average maximum force of pure Polyester was lower than the optimized composite.

Secondly, the average maximum stress of pure Polyester and the optimized composite was 24.50 N/mm2 and 40.77 N/mm2 respectively. This shows that the average maximum stress of pure Polyester was lower than the optimized composite.

Thirdly, the average maximum strain of pure Polyester and the optimized composite was 3.51% and 4.02% respectively. This shows that the average maximum strain of pure Polyester was lower than the optimized composite.

Overall, the mechanical properties of the composite have improved due to the addition of MWCNT to Polymer Resin. This is because MWCNT has a very high mechanical strength (Ma et al., 2010). Hence, this result can be accepted as valid.

4.3.2 Thermal Gravimetric Analysis (TGA)

Figure 4.5 shows the Thermal Gravimetric Analysis (TGA) results for both pure Polyester and the optimized composite (Polyester + 0.6wt % MWCNT) sample. In this analysis, TGA studies of samples were carried-out in a compressed air atmosphere on a thermal analyser at a heating rate of 10ºC/min. The compressed air was introduced to the system by 60:40 ratios and was heated from room temperature to 700ºC. 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.

From the figure, it can be seen that the pure Polyester sample starts to degrade after heating around 100ºC whereas the sample of 0.6wt % MWCNT starts to degrade only after heating around 200ºC. This shows that the optimized composite has higher thermal stability.

Secondly, it can be seen from the figure that both pure Polyester and 0.6wt % MWCNT sample has degraded completely before it even reaches the maximum temperature of 700ºC. However, the 0.6wt % MWCNT sample took even higher temperature to be

degraded when compared to Pure Polyester sample. This also shows the composite with 0.6wt % MWCNT has higher thermal stability.

As a conclusion, the addition of MWCNT into the composite could improve the thermal stability of the overall composite. This statement agrees with Aurilia et al. (2012) finding that MWCNT has high thermal strength. Hence, this result can be taken as valid.

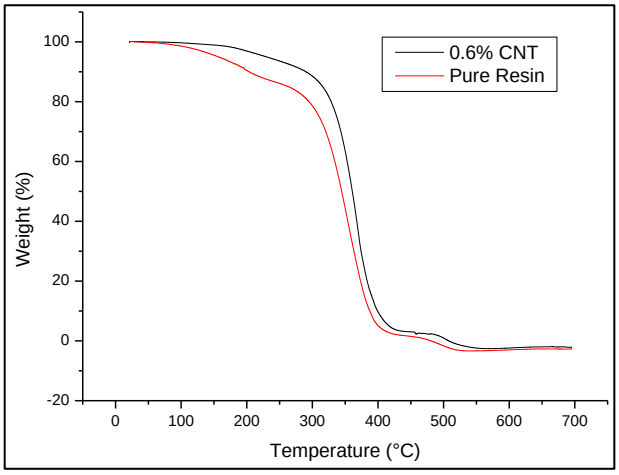

Figure 4.5: Thermal Gravimetric Analysis (TGA) Results for both Pure Polyester & Optimized Composite Sample

4.3.3 Fourier Transform Infrared Spectroscopy (FTIR)

Figure 4.6 shows the FTIR results for pure Polyester sample. From the figure, the peaks are at the wavenumbers of 606, 2360, 2500, 2850, 3170, 3570 and 3730 cm^{-1}. Some of the peaks can be identified by referring to Table 4.3. For instance, at 2500cm^{-1}, 2850cm^{-1} and 3570cm^{-1} the absorption characteristics are Carboxylic Acid O-H Stretch, Alkyl C-H Stretch and Amide N-H Stretch.

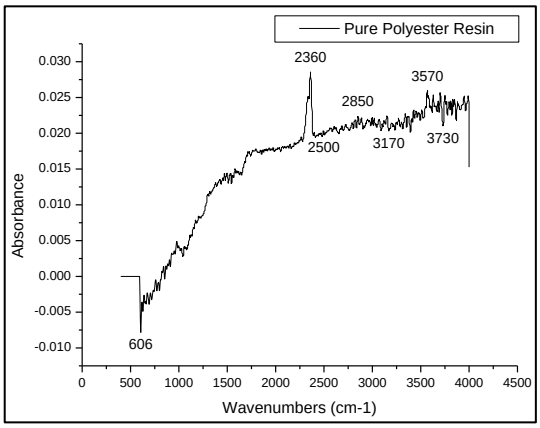

Figure 4.6: FTIR Result for Pure Polyester Sample

Figure 4.7 shows the FTIR results for the optimized composite (0.6wt % MWCNT). From the figure, the peaks are at the wavenumbers of 669, 2330, 3190, 3640, 3820 and 3980 cm^{-1}. The peaks were identified by referring to Table 4.3. For instance, at 3640cm^{-1} the absorption characteristics are Amide N-H Stretch.

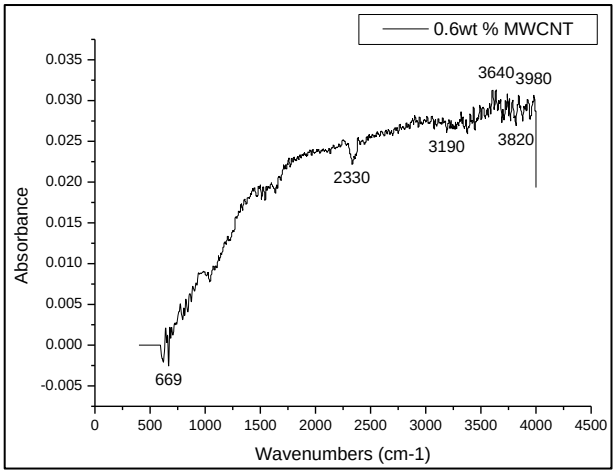

Figure 4.7: FTIR Result for Optimized Composite (Polyester + 0.6wt % MWCNT)

Table 4.3: IR Absorptions

Functional Group	*Characteristic Absorption(s)* (cm^{-1})
Alkyl C-H Stretch	2950 - 2850 (m or s)
Alkenyl C-H Stretch Alkenyl C=C Stretch	3100 - 3010 (m) 1680 - 1620 (v)
Alkynyl C-H Stretch Alkynyl C≡C Stretch	~3300 (s) 2260 - 2100 (v)
Aromatic C-H Stretch Aromatic C-H Bending Aromatic C=C Bending	~3030 (v) 860 - 680 (s) 1700 - 1500 (m,m)
Alcohol/Phenol O-H Stretch	3550 - 3200 (broad, s)
Carboxylic Acid O-H Stretch	3000 - 2500 (broad, v)
Amine N-H Stretch	3500 - 3300 (m)
Nitrile C≡N Stretch	2260 - 2220 (m)
Aldehyde C=O Stretch Ketone C=O Stretch Ester C=O Stretch Carboxylic Acid C=O Stretch Amide C=O Stretch	1740 - 1690 (s) 1750 - 1680 (s) 1750 - 1735 (s) 1780 - 1710 (s) 1690 - 1630 (s)
Amide N-H Stretch	3700 - 3500 (m)

(Source: Web Spectra, 2000)

Finally, the combined FTIR result for both pure Polyester and optimized composite sample are represented in Figure 4.8.

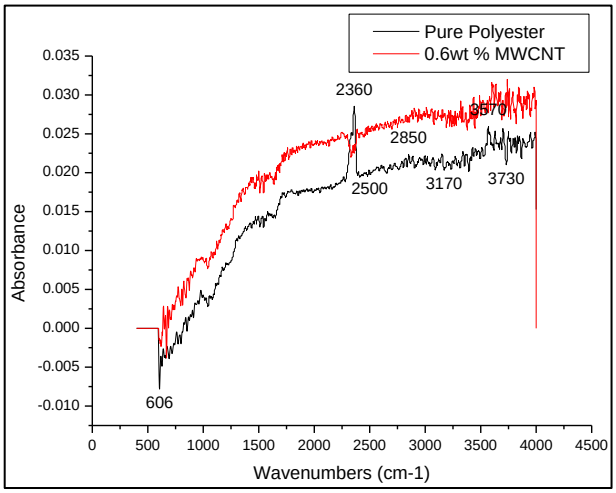

Figure 4.8: Combined FTIR Result for Both Samples

4.3.4 Summary

As a conclusion, a well improved MWCNT reinforced Polyester composite was produced. The optimum weight percentage of the composite was 0.6wt %, with the usage of solvent and the optimum sonication time that was obtained is 1.5 hour. The produced composite was successfully characterized and tested using Tensile Strength (TS), Thermal Gravimetric Analysis (TGA) and Fourier Transform Infrared Spectroscopy (FTIR).

5 CONCLUSION

5.1 Conclusion

This study focuses on the production and characterization of Hydroxyl functional Multi-Walled Carbon Nanotube (MWCNT) reinforced Polyester composite. The optimum weight percentage obtained was 0.6wt % MWCNT with solvent and the optimum sonication time obtained was 1.5 hour. The composite was then characterized using Tensile Strength (TS) Test, Thermal Gravimetric Analysis (TGA), and Fourier Transform Infrared Spectroscopy (FTIR).

5.2 Recommendations

The production of MWCNT can be explored more in future studies by three different ways. Firstly, different types of CNT such as Single-Walled, Double-Walled or surface modified CNTs could be tested and compared with this result. Secondly, the viscosity and dispersion could be compared by using other organic solvents such as toluene, chloroform, and DMF. Finally, the work can also be explored by replacing Polyester Resin with other polymeric material such as Epoxy Resin.

5.3 Future work

The research carried in this project is currently being expanded using other types of MWCNT such as Carboxyl functional MWCNT and non-functional MWCNT by Mr A.K.M. Moshiul Alam (PhD student). Finally, it is hoped that the study of MWCNT reinforced Polyester could be utilized in many applications in near future.

REFERENCES

Agnihotri, P., Basu, S., & Kar, K. K. (2011). Effect of carbon nanotube length and density on the properties of carbon nanotube-coated carbon fiber/polyester composites. *Carbon 49*, 3098-3106.

Al-Saleh, M. H., & Sundararaj, U. (2011). Review of the mechanical properties of carbon nanofiber/polymer composites. *Composites: Part A*, *42*, 2126–2142.

Aurilia, M., Sorrentino, L., & Iannace, S. (2012). Modelling physical properties of highly crystallized polyester reinforced with multiwalled carbon nanotubes. *European Polymer Journal*, *48*, 26–40.

Callister, W. D., & Rethwisch, D. G. (2008). *Fundamentals of materials science and engineering: An integrated approach*. Hoboken, NJ: John Wiley & Sons.

Che, J., Cagin, T., & Goddard, W. A. (2000). Thermal conductivity of carbon nanotubes.*Nanotechnology*, *11*, 65-69.

Chua, T. P., Mariatti, M., Azizan, A., & Rashid, A. A. (2010). Effects of surface-functionalized multi-walled carbon nanotubes on the properties of poly(dimethyl siloxane) nanocomposites. *Composites Science & Technology*, *70*, 671-677.

Dresselhaus, M. S., Dresselhaus, G., & Avouris, P. H. (2001). *Carbon nanotubes:synthesis, structure, properties, and applications*. Berlin: Springer.

Dresselhaus, M. S., Dresselhaus, G., & Saito, R. (1995). Physics of carbon nanotubes.*Carbon*, *33*, 883–891.

Du , J. H., Bai, J., & Cheng, H. M. (2007). The present status and key problems of carbon nanotube based polymer composites. *Express Polymer Letter*, *1*, 253–273.

Dumee, L. F., Sears, K., Marmiroli, B., Amenitsch, H., Duan, X., Lamb, R., Buso, D., Huynh, C., Hawkins, S., Kentish, S., Duke, M., Gray, S., Innocenzi, P., Hill, A. J., & Falcaro, P. (2013). A high volume and low damage route to hydroxyl functionalization of carbon nanotubes using hard X-ray lithography. *Carbon*, *51*, 427-434.

Ebbesen, T. W., Lezec, H. J., Hiura, H., Bennett,, J. W., Ghaemi, H. F., & Thio, T. (1996). Electrical conductivity of individual carbon nanotubes. *Nature*, *382*(6586), 54-56.

Esawi , A. M., & Farag , M. M. (2007). Carbon nanotube reinforced composites: potential and current challenges. *Materials & Design*, *28*, 2394–2401.

Fink, J. K. (2013). Unsaturated Polyester Resins. In *Reactive Polymers Fundamentals and Applications: A Concise Guide to Industrial Polymers* (2nd ed., pp. 1-48). Burlington: Elsevier Science.

Geng, H. Z., Rosen, R., Zheng, B., Shimoda, H., Fleming, L., Liu, J., & Zhou, O. (2002). Fabrication and Properties of Composites of Poly(ethylene oxide) and Functionalized Carbon Nanotubes. *Advance Material*, *14*(19), 1387–1390.

Gojny, F. H., & Schulte, K. (2004). Functionalization effect on the thermo-mechanical behavior of multiwalled carbon nanotube/epoxy composites. *Composites Science and Technology*,*34*, 2303–2308.

Gojny, F. H., Wichmann, M., Fiedler, B., & Schulte, K. (2005). Influence of different carbon nanotubes on the mechanical properties of epoxy matrix composites-a comparative study.*Composites Science & Technology*, *65*, 2300–2313.

Graham, S. R., Hodgson, R., Vechot, L., & Essa, M. I. (2011). Calorimetric studies on the thermal stability of methyl ethyl ketone peroxide (MEKP) formulations. *Process Safety and Environmental Protection*, *89*, 424–433.

Hilding, J., Grulke, E. A., Zhang, Z. G., & Lockwood, F. (2003). Dispersion of Carbon Nanotubes in Liquids. Journal of Dispersion Science and Technology, 24(1), 1–41.

Huang, F. M., Yur, S. W., & Chuang, M. Z. (2011). *Method for extending shelf life of vinyl ester resin or unsaturated polyester resin* (US Patent 8048965). Assigned to Swancor Inc. Co. Ltd.

Iijima, S. (1991). Helical microtubules of graphitic carbon. *Nature*, *354*, 56–58.

Jia, X. L., Zhang, Q., Huang, J. Q., Zheng, C., Qian, W. Z., & Wei, F. (2012). The direct dispersion of granular agglomerated carbon nanotubes in bismaleimide by high pressure homogenization for the production of strong composites. *Powder Technology*, *217*, 477–481.

Jung, J. M., Lee, D. H., & Cho, Y. I. (2013). Non-Newtonian standard viscosity fluids.*International Communications in Heat and Mass Transfer*, *49*, 1-4.

Kim, J. Y., Kim, D. K., & Kim, S. H. (2009). Effect of modified carbon nanotube on physical properties of thermotropic liquid crystal polyester nanocomposites. *European Polymer Journal*, *49*, 316–324.

Kinloch, I. A., Roberts, S. A., & Windle, A. H. (2002). A rheological study of concentrated aqueous nanotube dispersions. *Polymer*, *43*, 7483–7491

Kohlmeyer, R. R., Javadi, A., Pradhan, B., Pilla, S., Setyowati, K., Chen, J., & Gong, S. (2009). Electrical and dielectric properties of hydroxylated carbon nanotube−elastomer composites. The Journal of Physical Chemistry C, 113, 17626–17629.

Kosar, V., & Gomzi, Z. (2010). Crosslinking of an unsaturated polyester resin in the mould: Modelling and heat transfer studies. *Applied Mathematical Modelling*, *34*, 1586–1596.

Liu, C. X., & Choi, J. W. (2012). Improved Dispersion of Carbon Nanotubes in Polymers at High Concentrations. Nanomaterials, 2, 329-347.

Liu, T., Phang, I. Y., Shen, L., Chow, S. Y., & Zhang, W. D. (2004). Morphology and Mechanical Properties of Multiwalled Carbon Nanotubes Reinforced Nylon-6 Composites. *Macromolecules*, *37*, 7214-7222.

Lourie, O., Cox, D. M., & Wagner, H. D. (1998). Buckling and Collapse of Embedded Carbon Nanotubes. *Physical Review Letters*, *81*, 1638-1641.

Ma, H., Zeng, J., Realff, M. L., Kumar, S., & Schiraldi , D. A. (2003). Processing, structure, and properties of fibers from polyester/carbon nanofiber composites. *Computer Science Technology*, *63*(11), 1617–1628.

Ma, P. C., Siddiqui, N. A., Marom, G., & Kim, J. K. (2010). Dispersion and functionalization of carbon nanotubes for polymer-based nanocomposites: A review. *Composites: Part A*,*41*, 1345–1367.

Patel, R. K., Bhattacharya, B., & Basu, S. (2008). Effect of interphaseproperties on the damping response of polymer nano-composites. *Mechanics Research Communications*,*35*(1-2), 115–125.

Paul, D. R., & Robeson, L. M. (2008). Polymer nanotechnology: nanocomposites. *Polymer*,*49*(15), 3187–3204.

Reynaud, E., Gauthier, C., & Perez, J. (1999). Nanophases in polymers. *Rev Metall*, *96*, 169–176.

Ring, K. L., Schlag, S., & Toki, G. (2002). *Report Unsaturated Polyester Resins, in: Chemical Economics Handbook (CEH)*. Retrieved from SRI Consulting, a Division of Access Intelligence, Menlo Park, CA website: http://ceh.sric.sri.com/

Seyhan, A. T., Gojny, F. H., Tanoglu, M., & Schulte, K. (2007). Critical aspects related to processing of carbon nanotube/unsaturated thermoset polyester nanocomposites.*European Polymer Journal*, *43*, 374–379.

Shokrieh, M. M., Saeedi, A., & Chitsazzadeh, M. (2014). Evaluating the effects of multi-walled carbon nanotubes on the mechanical properties of chopped strand mat/polyester composites. *Materials & Design*, *56*, 274–279.

Shu-quan, L., Chun-yan, J., Yan, T., Yong, Z., Jie, Z., & An-qiang, P. (2007). Mechanical and electrical properties of carbon nanotube reinforced epoxide resin composites. *Transaction of Nonferrous Metals Society of China*, *17*, 675–679.

Spitalsky, Z., Tasis, D., Papagelis, K., & Galiotis, C. (2010). Carbon nanotube–polymer composites: Chemistry, processing, mechanical and electrical properties. *Progress in Polymer Science*, *35*, 357–401.

Thostenson, E. T., Li, C., & Chou, T. W. (2005). Nanocomposites in context. *Composites Science and Technology*, *65*(3-4), 491-516.

Velasco-Santos, V., Martinez-Hernandez, A. L., & Castano, V. M. (2005). Carbon nanotube-polymer nanocomposites: The role of interfaces. *Composite Interfaces*, *11*(89), 567–586. Retrieved from www.vsppub.com

Web Spectra: Table of IR Absorptions. (2000). Retrieved May 29, 2014, from http://www.chem.ucla.edu/~webspectra/irtable.html

Wong, M., Paramsothy, M., Xu, X. J., Ren , Y., Li, S., & Liao, K. (2003). Physical interactions at carbon nanotube–polymer interface. *Polymer*, *44*(25), 7757–7764.

APPENDIX A: Results for Different Weight Percentage of MWCNT When No Solvent Was Used

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	636.18	0.21	2.19	671.30	0.22	1.99	609.54	0.21	2.09	639.01	0.21	0.34
10.00	20.80	632.90	2.14	21.90	655.10	2.16	19.90	611.10	2.11	20.87	633.03	2.14	3.40
20.00	38.30	569.90	3.86	38.40	599.90	3.89	35.50	533.90	3.81	37.40	567.90	3.85	6.80
30.00	54.00	535.90	5.44	54.90	565.40	5.48	52.90	500.90	5.39	53.93	534.07	5.44	10.20
40.00	68.80	513.60	6.99	69.10	539.90	7.02	64.90	489.90	6.56	67.60	514.47	6.86	13.60
50.00	83.30	497.90	8.47	89.90	518.90	8.44	81.90	467.10	8.44	85.03	494.63	8.45	17.00
60.00	95.80	487.40	9.95	97.60	488.90	9.90	92.30	452.90	9.90	95.23	476.40	9.92	20.40
70.00	-	-	-	-	-	-	99.70	432.10	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	640.69	0.22	1.99	621.83	0.21	2.13	677.91	0.22	2.06	646.81	0.21	0.34
10.00	20.70	640.80	2.15	19.90	621.10	2.10	21.30	666.90	2.17	20.63	642.93	2.14	3.40
20.00	38.20	571.10	3.85	37.60	554.90	3.81	39.00	599.90	3.88	38.27	575.30	3.85	6.80
30.00	54.20	539.90	5.49	54.10	519.90	5.44	55.60	579.00	5.51	54.63	546.27	5.48	10.20
40.00	69.50	517.40	7.02	69.00	499.90	6.99	73.90	536.70	7.05	70.80	518.00	7.02	13.60
50.00	84.10	502.70	8.54	82.90	478.90	8.52	88.10	523.90	8.58	85.03	501.83	8.55	17.00
60.00	98.80	493.40	10.10	97.50	477.90	10.00	99.90	500.90	10.10	98.73	490.73	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	666.91	0.23	2.09	631.47	0.22	2.22	691.81	0.23	2.15	663.40	0.22	0.34
10.00	21.40	659.90	2.25	20.90	633.90	2.21	22.20	681.10	2.25	21.50	658.30	2.24	3.40
20.00	40.20	599.90	4.07	39.80	571.90	4.05	43.30	613.90	4.09	41.10	595.23	4.07	6.80
30.00	56.90	566.90	5.74	55.50	533.90	5.73	58.90	599.90	5.75	57.10	566.90	5.74	10.20
40.00	72.80	542.90	7.37	71.10	521.90	7.36	74.90	556.90	7.39	72.93	540.57	7.37	13.60
50.00	88.20	524.30	8.95	88.00	514.10	8.92	91.10	534.10	8.99	89.10	524.17	8.95	17.00
60.00	98.10	511.30	9.92	97.20	499.90	9.90	99.90	523.90	10.00	98.40	511.70	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	690.06	0.24	2.25	712.95	0.23	2.11	682.92	0.23	2.21	695.31	0.23	0.34
10.00	22.70	686.90	2.35	22.50	699.10	2.34	21.10	675.90	2.33	22.10	687.30	2.34	3.40
20.00	41.10	613.40	4.17	43.90	645.10	4.19	40.90	600.90	4.14	41.97	619.80	4.17	6.80
30.00	57.80	574.90	5.84	59.10	575.10	5.99	56.30	573.90	5.81	57.73	574.63	5.88	10.20
40.00	73.40	547.40	7.40	74.70	566.90	7.45	69.90	533.20	7.29	72.67	549.17	7.38	13.60
50.00	88.20	526.70	8.92	89.10	532.10	9.04	80.90	522.90	8.67	86.07	527.23	8.88	17.00
60.00	99.30	521.10	10.10	99.90	523.10	10.10	89.90	500.90	9.15	96.37	515.03	9.78	20.40
70.00	-	-	-	-	-	-	99.80	489.10	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	699.12	0.24	2.31	708.90	0.24	2.41	718.57	0.24	2.33	708.86	0.24	0.34
10.00	22.80	689.90	2.36	23.10	700.10	2.39	24.10	713.90	2.41	23.33	701.30	2.39	3.40
20.00	42.20	629.90	4.26	43.30	641.90	4.29	44.40	655.90	4.31	43.30	642.57	4.29	6.80
30.00	59.60	590.90	6.02	60.90	610.90	6.15	61.90	621.40	6.19	60.80	607.73	6.12	10.20
40.00	76.00	566.10	7.70	79.90	586.90	7.75	83.90	599.90	7.78	79.93	584.30	7.74	13.60
50.00	88.90	547.10	9.30	89.90	567.90	9.33	90.10	588.10	9.95	89.63	567.70	9.53	17.00
60.00	98.30	531.40	10.00	98.90	551.10	10.10	99.90	572.20	10.10	99.03	551.57	10.07	20.40
70.00	-	-	-	-	-	-	-	-	-	-	-	-	23.80
80.00	-	-	-	-	-	-	-	-	-	-	-	-	27.20

f) 0.6% CNT (0.30 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	1.30	0.00	0.16	0.50	0.00	0.05	0.20	0.00	0.01	0.67	0.00	0.07	0.00
1.00	2.52	791.27	0.26	2.41	775.76	0.26	2.63	823.24	0.26	2.52	796.76	0.26	0.34
10.00	25.20	758.80	2.59	24.10	739.90	2.56	26.30	789.90	2.61	25.20	762.87	2.59	3.40
20.00	45.00	714.20	4.54	44.90	699.10	4.51	45.40	735.40	4.59	45.10	716.23	4.55	6.80
30.00	60.70	664.40	6.17	59.70	634.90	6.14	61.00	689.90	6.21	60.47	663.07	6.17	10.20
40.00	68.30	612.30	6.95	65.90	589.90	6.90	71.80	635.50	7.05	68.67	612.57	6.97	13.60
50.00	82.40	589.10	8.34	80.90	565.40	8.29	83.90	614.40	8.55	82.40	589.63	8.39	17.00
60.00	96.70	562.00	9.75	94.90	531.00	9.45	99.90	572.90	10.10	97.17	555.30	9.77	20.40
70.00	-	-	-	-	-	-	-	-	-	-	-	-	23.80
80.00	-	-	-	-	-	-	-	-	-	-	-	-	27.20

g) 0.7% CNT (0.35 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	0.00	0.00	0.00	0.40	0.00	0.04	0.10	0.00	0.01	0.17	0.00	0.02	0.00
1.00	2.36	731.70	0.24	2.55	766.58	0.25	2.45	683.91	0.25	2.45	727.40	0.25	0.34
10.00	23.60	699.90	2.44	25.50	739.90	2.51	24.50	650.90	2.49	24.53	696.90	2.48	3.40
20.00	41.00	654.30	4.29	42.20	676.90	4.31	43.30	599.90	4.32	42.17	643.70	4.31	6.80
30.00	59.90	599.60	5.97	62.30	633.20	6.02	63.30	549.90	6.00	61.83	594.23	6.00	10.20
40.00	73.90	543.30	7.41	75.60	576.40	7.45	75.90	499.90	7.43	75.13	539.87	7.43	13.60
50.00	87.60	522.90	8.85	91.10	565.90	9.02	89.90	467.10	9.00	89.53	518.63	8.96	17.00
60.00	94.40	499.90	9.57	99.90	523.20	10.10	99.70	443.00	10.00	98.00	488.70	9.89	20.40
70.00	-	-	-	-	-	-	-	-	-	-	-	-	23.80
80.00	-	-	-	-	-	-	-	-	-	-	-	-	27.20

h) 0.8% CNT (0.40 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	0.05	0.20	0.00	0.01	0.40	0.00	0.04	0.23	0.00	0.03	0.00
1.00	2.94	674.47	0.30	2.63	702.68	0.26	2.55	724.98	0.25	2.71	700.71	0.27	0.34
10.00	29.40	649.90	2.97	26.30	678.90	2.61	25.50	699.90	2.51	27.07	676.23	2.70	3.40
20.00	45.10	599.90	4.55	45.40	622.20	4.59	42.20	644.40	4.31	44.23	622.17	4.48	6.80
30.00	62.30	520.30	6.21	61.00	543.30	6.21	62.30	567.90	6.02	61.87	543.83	6.15	10.20
40.00	77.50	499.90	7.87	71.80	522.90	7.05	75.60	544.40	7.45	74.97	522.40	7.46	13.60
50.00	91.90	470.40	9.42	83.90	499.90	8.55	91.10	523.30	9.02	88.97	497.87	9.00	17.00
60.00	98.30	444.30	10.10	99.90	460.10	10.10	99.90	480.90	10.10	99.37	461.77	10.10	20.40
70.00	-	-	-	-	-	-	-	-	-	-	-	-	23.80
80.00	-	-	-	-	-	-	-	-	-	-	-	-	27.20

APPENDIX B: Results for Different Weight Percentage of MWCNT When Solvent Was Used

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	880.78	0.21	1.92	908.28	0.22	1.72	852.36	0.20	1.84	880.47	0.21	0.34
10.00	18.70	863.80	2.09	19.20	893.30	2.15	17.20	823.30	2.03	18.37	860.13	2.09	3.40
20.00	29.80	743.90	2.98	30.10	777.70	3.02	28.30	713.30	2.93	29.40	744.97	2.98	6.80
30.00	38.50	689.90	3.89	39.00	702.20	3.91	36.50	655.50	3.85	38.00	682.53	3.88	10.20
40.00	45.50	612.20	4.56	47.60	643.30	4.71	44.10	589.90	4.52	45.73	615.13	4.60	13.60
50.00	59.90	560.90	6.01	62.10	599.90	6.23	57.50	532.20	5.95	59.83	564.33	6.06	17.00
60.00	71.20	499.90	7.02	72.90	523.30	7.05	69.90	467.70	6.99	71.33	496.97	7.02	20.40
70.00	88.30	467.20	8.96	89.90	489.90	9.01	86.50	411.10	8.91	88.23	456.07	8.96	23.80
80.00	96.70	421.10	9.96	99.80	454.30	10.10	95.50	378.90	9.93	97.33	418.10	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	916.69	0.22	2.15	961.26	0.22	2.11	882.59	0.22	2.13	920.18	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	645.50	4.73	52.00	678.80	5.23	46.70	612.20	4.71	48.87	645.50	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	523.30	7.01	74.40	555.50	7.15	73.30	499.90	6.99	72.93	526.23	7.05	20.40
70.00	90.00	500.90	9.02	89.90	519.90	9.01	88.20	465.50	8.77	89.37	495.43	8.93	23.80
80.00	98.80	476.50	9.95	99.90	478.60	10.10	97.60	433.20	9.89	98.77	462.77	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	972.68	0.20	2.14	1010.10	0.22	1.88	930.20	0.21	1.91	970.99	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	1006.55	0.20	2.15	1024.27	0.22	2.13	976.70	0.22	1.99	1002.51	0.21	0.34
10.00	16.90	967.70	2.02	21.50	984.40	2.19	21.30	932.20	2.18	19.90	961.43	2.13	3.40
20.00	28.50	865.50	2.93	32.20	899.90	3.09	30.50	839.90	3.04	30.40	868.43	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	800.37	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	711.97	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	681.40	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	603.27	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	541.40	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	1040.73	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

f) 0.6% CNT (0.30 g CNT; 7.50 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	0.10	0.00	0.01	0.20	0.00	0.01	0.10	0.00	0.01	0.13	0.00	0.01	0.00
1.00	2.15	1086.33	0.22	1.71	1124.99	0.20	1.86	1050.50	0.21	1.91	1087.27	0.21	0.34
10.00	21.50	1040.90	2.19	17.10	1078.80	2.03	18.60	1011.20	2.09	19.07	1043.63	2.10	3.40
20.00	32.20	954.40	3.09	28.10	985.50	2.95	29.90	923.30	2.98	30.07	954.40	3.01	6.80
30.00	41.10	887.20	4.01	36.40	922.20	3.86	38.80	845.50	3.89	38.77	884.97	3.92	10.20
40.00	52.00	799.90	5.23	44.40	834.30	4.52	46.60	756.60	4.58	47.67	796.93	4.78	13.60
50.00	65.30	734.40	6.55	57.80	765.50	5.95	60.10	699.90	6.13	61.07	733.27	6.21	17.00
60.00	74.40	676.60	7.15	70.00	699.90	7.01	70.90	643.30	7.06	71.77	673.27	7.07	20.40
70.00	89.90	621.10	9.01	87.10	656.60	8.92	87.70	599.90	8.99	88.23	625.87	8.97	23.80
80.00	99.90	579.80	10.10	96.20	599.70	9.95	97.10	543.30	9.97	97.73	574.27	10.01	27.20

g) 0.7% CNT (0.35 g CNT; 8.75 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	0.50	0.00	0.05	0.20	0.00	0.01	0.20	0.00	0.01	0.30	0.00	0.02	0.00
1.00	1.91	988.45	0.21	1.92	955.10	0.22	1.72	931.14	0.20	1.85	958.23	0.21	0.34
10.00	19.10	959.90	2.08	19.20	929.90	2.15	17.20	899.90	2.03	18.50	929.90	2.09	3.40
20.00	29.90	857.70	2.98	30.10	822.20	3.02	28.30	799.90	2.93	29.43	826.60	2.98	6.80
30.00	38.30	782.20	3.89	39.00	754.40	3.91	36.50	733.30	3.85	37.93	756.63	3.88	10.20
40.00	48.80	672.20	4.61	47.60	643.30	4.71	44.10	611.10	4.52	46.83	642.20	4.61	13.60
50.00	63.30	621.90	6.24	62.10	589.90	6.23	57.50	556.60	5.95	60.97	589.47	6.14	17.00
60.00	72.20	557.10	7.09	72.90	533.30	7.05	69.90	501.10	6.99	71.67	530.50	7.04	20.40
70.00	87.30	521.70	8.91	89.90	489.90	9.01	86.50	456.60	8.91	87.90	489.40	8.94	23.80
80.00	99.80	487.90	10.10	99.80	465.50	10.10	95.50	433.30	9.93	98.37	462.23	10.04	27.20

h) 0.8% CNT (0.40 g CNT; 10.00 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	0.10	0.00	0.01	0.20	0.00	0.01	0.10	0.00	0.01	0.13	0.00	0.01	0.00
1.00	1.81	935.45	0.20	2.13	915.38	0.22	2.15	957.27	0.22	2.03	936.03	0.21	0.34
10.00	18.10	901.10	2.03	21.30	879.90	2.18	21.50	923.30	2.19	20.30	901.43	2.13	3.40
20.00	28.80	821.10	2.93	30.50	799.90	3.04	32.20	845.50	3.09	30.50	822.17	3.02	6.80
30.00	36.60	732.20	3.85	37.90	702.20	3.86	41.10	745.50	4.01	38.53	726.63	3.91	10.20
40.00	44.00	632.20	4.52	47.90	611.90	4.73	52.00	654.40	5.23	47.97	632.83	4.83	13.60
50.00	56.60	589.50	5.95	62.00	556.60	6.23	65.30	610.10	6.55	61.30	585.40	6.24	17.00
60.00	68.80	502.20	6.99	71.10	487.70	7.01	74.40	523.30	7.15	71.43	504.40	7.05	20.40
70.00	87.30	488.70	8.91	90.00	456.60	9.02	89.90	501.10	9.01	89.07	482.13	8.98	23.80
80.00	96.70	456.50	9.93	98.80	423.30	9.95	99.90	478.80	10.10	98.47	452.87	9.99	27.20

APPENDIX C: Comparison Results for Different Weight Percentage of MWCNT

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	0.6% CNT	0.7% CNT	0.8% CNT	0.1% CNT	0.2% CNT	0.3% CNT	0.4% CNT	0.5% CNT	0.6% CNT	0.7% CNT	0.8% CNT
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.34	639.01	646.81	663.40	695.31	708.86	796.76	727.40	700.71	880.47	920.18	970.99	1002.51	1040.73	1087.27	958.23	936.03
3.40	633.03	642.93	658.30	687.30	701.30	762.87	696.90	676.23	860.13	902.10	933.90	961.43	1000.90	1043.63	929.90	901.43
6.80	567.90	575.30	595.23	619.80	642.57	716.23	643.70	622.17	744.97	783.27	835.53	868.43	914.40	954.40	826.60	822.17
10.20	534.07	546.27	566.90	574.63	607.73	663.07	594.23	543.83	682.53	729.77	762.43	800.37	844.40	884.97	756.63	726.63
13.60	514.47	518.00	540.57	549.17	584.30	612.57	539.87	522.40	615.13	645.50	678.47	711.97	743.87	796.93	642.20	632.83
17.00	494.63	501.83	524.17	527.23	567.70	589.63	518.63	497.87	564.33	600.70	634.07	681.40	696.07	733.27	589.47	585.40
20.40	476.40	490.73	511.70	515.03	551.57	555.30	488.70	461.77	496.97	526.23	555.63	603.27	637.37	673.27	530.50	504.40
23.80									456.07	495.43	505.87	541.40	582.10	625.87	489.40	482.13
27.20									418.10	462.77	465.50	499.90	547.37	574.27	462.23	452.87

APPENDIX D: Results for Different Sonication Time of Composite

a) 0.5 hour

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	925.88	0.22	1.87	866.31	0.20	2.01	961.44	0.23	1.96	917.88	0.22	0.34
10.00	19.90	887.20	2.15	18.70	845.50	1.98	20.10	921.10	2.32	19.57	884.60	2.15	3.40
20.00	28.40	799.90	2.99	27.30	734.40	2.88	29.90	843.30	2.90	28.53	792.53	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	588.90	4.21	41.00	532.20	4.17	45.50	632.20	4.32	42.87	584.43	4.23	13.60
50.00	59.90	511.90	5.55	57.80	476.60	5.51	61.10	543.30	5.87	59.60	510.60	5.64	17.00
60.00	70.90	487.40	7.60	69.10	421.10	7.55	73.20	532.20	7.71	71.07	480.23	7.62	20.40
70.00	88.20	401.10	8.98	87.60	376.60	8.95	89.90	445.50	9.03	88.57	407.73	8.99	23.80
80.00	99.80	378.10	10.00	95.60	343.30	9.87	99.90	423.20	10.10	98.43	381.53	9.99	27.20

b) 1.0 hour

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	1086.33	0.20	1.88	1138.25	0.20	1.91	1031.79	0.21	1.83	1085.46	0.20	0.34
10.00	16.90	1040.90	2.02	18.80	1099.20	2.01	19.10	1011.10	2.08	18.27	1050.40	2.04	3.40
20.00	28.50	954.40	2.93	30.10	989.20	3.29	29.90	921.10	2.98	29.50	954.90	3.07	6.80
30.00	36.30	887.20	3.85	39.90	921.10	3.94	38.30	843.30	3.89	38.17	883.87	3.89	10.20
40.00	43.90	799.90	4.53	48.20	834.40	4.56	48.80	755.50	4.61	46.97	796.60	4.57	13.60
50.00	57.20	734.40	5.95	65.50	766.60	6.55	63.30	744.50	6.24	62.00	748.50	6.25	17.00
60.00	70.10	676.60	7.00	78.90	703.30	7.50	72.20	699.80	7.09	73.73	693.23	7.20	20.40
70.00	87.70	621.10	8.92	89.90	643.30	9.02	87.30	645.30	8.91	88.30	636.57	8.95	23.80
80.00	97.40	579.80	10.00	98.40	599.50	9.89	99.80	589.90	10.10	98.53	589.73	10.00	27.20

c) 1.5 hour

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	1138.47	0.20	2.15	1161.45	0.22	2.15	1100.26	0.22	2.01	1133.40	0.21	0.34
10.00	17.20	1086.60	2.03	21.50	1100.10	2.19	21.50	1045.50	2.19	20.07	1077.40	2.14	3.40
20.00	28.30	992.20	2.93	32.20	1020.30	3.09	32.20	954.40	3.09	30.90	988.97	3.04	6.80
30.00	36.50	922.90	3.85	41.10	945.50	4.01	41.10	889.30	4.01	39.57	919.23	3.96	10.20
40.00	44.10	843.30	4.52	52.00	876.60	5.23	52.00	799.90	5.23	49.37	839.93	4.99	13.60
50.00	57.50	776.30	5.95	65.30	799.10	6.55	65.30	743.30	6.55	62.70	772.90	6.35	17.00
60.00	69.90	701.10	6.99	74.40	732.20	7.15	74.40	676.60	7.15	72.90	703.30	7.10	20.40
70.00	86.50	654.40	8.91	89.90	675.40	9.01	89.90	612.20	9.01	88.77	647.33	8.98	23.80
80.00	95.50	583.30	9.93	99.90	602.20	10.10	99.90	543.30	10.10	98.43	576.27	10.04	27.20

d) 2.0 hour

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	1149.99	0.22	2.13	1092.15	0.22	1.92	1182.16	0.22	2.05	1141.43	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) 2.5 hour

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	1188.91	0.21	1.72	1093.46	0.20	2.13	1165.89	0.22	1.91	1149.42	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