EVALUATION OF ELECTROPHORETIC DEPOSITION OF MULTI-WALLED CARBON NANOTUBES ONTO CARBON FIBER

Patrick Renaldo Bin Richard

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PATRICK RENALDO BIN RICHARD

A dissertation submitted in partial fulfilment of requirement for the degree of Bachelor of Engineering with Honours

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To my beloved family and friends

and

thank you, God, for giving me the strength to keep going

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# ABSTRACT

The Fiber Reinforced Polymer Composite (FRPC) has been used widely in the structural application, however, the incorporation of nanoparticle such as multi-walled carbon nanotubes (MWCNT) can be utilized to further enhance the mechanical properties of the composites. The aim of this study is to compare the stability of MWCNT in distilled water (DW) and Dimethylformamide (DMF). The chosen incorporation method of deposition of MWCNT onto carbon fiber (CF) were electrophoretic deposition (EPD) due to the advantages of simple apparatus, short function time and low cost. Therefore, the effect of voltages and time were studied to obtain the optimal condition for deposition of MWCNT into CF. The stability of dispersed MWCNT in different types of medium were studied as the importance of the MWCNT to stay stable in the medium is required to attain homogeneous deposition. The UV-vis and colloidal stability test showed that DMF has better stability than DW in the long run. Scanning Electrode Microscopy (SEM) images showed that the best condition for the deposition of MWCNT onto CF were to be deposition time of 10 mins and applied voltage of 20 V. Therefore, the MWCNT dispersed in DW is still valid to be used as dispersing medium for EPD process as the time required for depositing MWCNT onto CF is short and the colloidal suspension is still stable within the time frame.

# ABSTRAK

Komposit Polimer Bertetulang Gentian (FRPC) telah digunakan secara meluas dalam aplikasi struktur, bagaimanapun, penggabungan nanopartikel seperti nanotube karbon multi-wall(MWCNT) dapat digunakan untuk meningkatkan sifat mekanik komposit. Tujuan kajian ini adalah untuk membandingkan kestabilan MWCNT dalam air suling (DW) dan Dimethylformamide (DMF). Kaedah penyatuan pemilihan MWCNT ke serat karbon (CF) adalah pemendapan electrophoretic (EPD) kerana kelebihan alat mudah, masa fungsi pendek dan kos rendah. Oleh itu, kesan voltan dan masa dikaji untuk mendapatkan keadaan optimum untuk pemendapan MWCNT ke dalam CF. Kestabilan MWCNT yang tersebar dalam pelbagai jenis media telah dikaji kerana kepentingan MWCNT untuk kekal stabil dalam medium diperlukan untuk mencapai pemendapan homogen. Ujian kestabilan UV-vis dan koloid menunjukkan bahawa DMF mempunyai kestabilan yang lebih baik daripada DW dalam jangka masa panjang. Pengimbasan Gambar Mikroskopi Elektroda (SEM) menunjukkan bahawa keadaan terbaik untuk pemendapan MWCNT ke CF adalah waktu pengendapan selama 10 minit dan voltan dikenakan 20 V. Oleh karena itu, MWCNT yang tersebar di DW masih berlaku untuk digunakan sebagai media penyebaran untuk proses EPD sebagai masa yang diperlukan untuk mendepositkan MWCNT ke CF adalah pendek dan penggantungan koloid masih stabil dalam tempoh masa.

# TABLE OF CONTENTS

# LIST OF TABLES

**Table** **Page**

# LIST OF FIGURES

**Figure** **Page**

# LIST OF ABBREVIATIONS

| | |
|---|---|
| BET | Brunauer-Emmett-Teller |
| CF | Carbon Fiber |
| CNT | Carbon Nanotube |
| CVD | Chemical Vapor Deposition |
| DD | Dipping Deposition Method |
| EPD | Electrophoretic Distribution |
| FTIR | Fourier Transform Infrared Spectroscopy |
| MWCNT | Multiwall Carbon Nanotubes |
| UV-Vis | Ultraviolet Visible |

# CHAPTER 1

# INTRODUCTION

## 1.1 Research Background

Fiber reinforced polymer (FRP) are a form of composite that the polymer matrix is reinforced by fibers. Whereby, composite materials are defined as material that are naturally occurring or fabricated of two or more constituent materials which significant different physical and chemical properties that remains to be separated within the finished product (Alberto, 2013). FRP are a type of composite which utilizes fiber materials to mechanically enhance mechanical properties of polymer matrix. The extend of the enhancement in mechanical and the FRP are affected by the types of fibers and matrix used, volume ratio of matrix and fibers, fiber length and orientation of within the matrix.

Reinforcement of the matrix apply when the composite exhibits enhancement of chemical and mechanical properties relative to the matrix alone. There are wide range of fibers used in fabrication of this composite which are generally can be classified as natural and synthetic fibers. Natural fibers are fibers that can be extracted from plants or animals direct while synthetic fibers are those that man-made. Alberto (2013), stated that the most commonly used fibers are glass and carbon fibers within a thermosetting polymer. FRP are favoured due to their benefits such as higher strength to weight ratio.

FRP are widely used in structures such as air frame, sports equipment, and automobile. Alberto (2013) stated that FRP are viewed to be an upgrade from the traditional infrastructure component such as steel and concrete in civil engineering and the application of this composite are continually growing to fields such as biomedical. A study stated that due to FRP's better mechanical performance, higher damage resistance, and lower thermal effects, FRP can provide longer service life than traditional materials provided that FRP were to be fabricated with high precision in designing, fabrication, and assemblage (Kutz, 2017). Carbon fiber reinforced polymers (CFRP) are considered to be one of the important structural materials in modern engineering application due to their excellent mechanical and chemical properties (Xian & Wang, 2018). In Table 1.1 shows example of applications of FRP in industry. It shows that this composite can be used to replace the traditional material used such as metal in parts of a ship to further reduce the cumulative mass of the ship.

There are many types of fabrication of FRP where they are ranging from simple set up and economically feasible to automated set up whereby requiring high investments (Agarwal et al., 2018). The researcher stated that in the inexpensive methods however, there are variable of properties that could result, as it is highly depending on the skills of the operator. A study shows that these variables of properties are due to defects formation in fabrication of FRP (Mehdikhani et al., 2018). The researcher defined the defects as cause of deviation of composite properties due to irregularities. The most important defect that required to be considered is void due to it is commonly occurred compared to other defects such as contamination and delamination (Liu & Chen, 2016). A study also shows that properties of composites are dependent on the performance and microstructure of interphase between fiber and matrix whereby the region where the fiber and matrix are mechanically and chemically combined (L. G. Tang & Kardos, 1997). The researcher noted that bonding between the fiber and matrix can provide better properties of the product produced and therefore surface modification of the structure of the surface of the fiber has been done in previous researches to improve the adhesion between the fiber and matrix. An example of the method to modify surface structure of a fiber is electrophoretic deposition (EPD).

Table 1.1 Application of FRP in industry

| Industry | Fiber reinforced polymer | Application |
|---|---|---|
| Aerospace | Carbon fiber reinforced polymer | Boeing A380<br>Tail planes<br>Tail cone<br>Un-pressurized fuselage<br>Outer flaps<br>Rear pressure bulkhead<br>Engine Cowlings<br>Flap track panels<br>Landing gear doors<br>Centre wing box<br>Wing<br>Outer flaps<br>Wing ribs<br>Upper deck floor beams |
| Automotive | Carbon fiber reinforced polymer | Car<br>Door frame pillar<br>Seat back<br>Fender support<br>Radiator core support<br>Hood<br>Roof<br>Door<br>Spoiler |
| Marine | Aramid fiber reinforced polymer | Kayak |
| | Glass fiber reinforced polymer | Maritime Naval Ship<br>Mine sweeper<br>Landing craft<br>Personnel boat<br>Sheathing of wood hulls<br>Submarine sonar dome<br>Submarine firs<br>Landing craft reconnaissance<br>Submarine non-pressure hull casing |

EPD used to deposit stable inorganic particles in a solvent called suspension onto substrate that are electrically conductive (Sarkar & Nicholson, 1996). Thomas et al., (2005) stated that this technique can produce controlled thickness and homogeneous microstructure of oxide coating on a wide range of substrate. According to Boccaccini et al. (2006), materials that undergo EPD process exhibits good microstructure homogeneity and high packing density. Electrophoretic distribution is a technique that involves two process. First step is the forcing of suspended particles to move towards electrode, and the second step is the collection of particles at one of the electrodes and forms coherent deposit. This process will only be effective when electric field is applied to the suspension, thus, the process is called electrophoresis.

According to Vandeperre & Biest (1999), electrophoretic deposition can be utilized to solids that are available in powder form that are less than 30µm or a colloidal suspension. Previous findings state that the process can be used for infiltration of porous materials and woven fiber preforms for composite production, laminated and graded free-standing objects, shape monolithic, and producing coatings. This research focuses on electrophoretic distribution of carbon nanotubes. Carbon nanotubes has unique characteristics such as possessing higher stiffness, higher aspects ratio and lower density (C. Li & Chou, 2003). According to Li and Chou (2003), they stated that these characteristics are favourable for fabricating a modern generation of composite.

## 1.2 Problem Statement

CNT has attracted researchers for range of potential applications, which includes utilizing them as filler for fiber reinforced composite. In general MWCNT, as they possess excellent mechanical, electrical, thermal properties (Thomas et al., 2005). However, Johann et al., (2006) stated that if MWCNT were to be combined with other material, it is important for them to be dispersed homogenously in the matrix. Previous studies showed that inhomogeneous and agglomerated CNT onto composite will not enhance the properties of the composite (Kuzumaki et al., 1998). The agglomeration of CNT are due to strong van der Waals forces between then carbon (Hawthorn, 1989). Therefore, the best method to deposit homogenous MWCNT will be EPD. Johann et al., (2006) stated that EPD is an advanced level efficient process to produce coating from colloidal suspension where the deposited material exhibits good microstructure homogeneity and high packing density.

To be able to utilize the deposition method, a colloidal suspension has to be prepared as study previously stated various solvent can be used to prepare MWCNT suspension for EPD, which includes DW and DMF (Pourgheysari et al., 2016). A study also shows that MWCNT are generally has poor compatibility with most materials due to their high total surface free area which leads the nanoparticle to be hydrophobic (Janas & Stando, 2017). This finding causes harder dispersion of MWCNT in aqueous based solution. The most common method to dispersed MWCNT in aqueous solution is by acid treatment. This causes introduction of functional groups of OH, COOH, and C=O onto the surface of MWCNT (F. Avilés et al., 2009a). However, for mechanical reinforcement of composite the aspect ratio required for MWCNT has to be large for adequate load transfer (Hou et al., 2008). Acid treatment causes shortening of MWCNT and thus reduces the properties of MWCNT and therefore will not enhance the properties of composite (Wan et al., 2005).

## 1.3 Objectives

The objective of this research are as follows;

1. To study the effects of applied voltages and deposition time on the deposition of multi-walled carbon nanotubes (MWCNT) onto carbon fiber (CF) surface.
2. To compare the stability of MWCNT in the organic and non-organic mediums.
3. To investigate the hardness properties of MWCNT/CF/Epoxy laminated surface

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Fiber Reinforced Polymer Composite

### 2.1.1 Introduction

Fiber reinforced polymer composite has been widely used as a primary load-bearing structure primarily in aerospace industry (Coleman et al., 2006). Carbon fiber are an example of a widely used as a fiber component in a composite due to their low densities and superior mechanical properties (Lin et al., 2009). The researchers stated that the fiber is used to improve the performance of polymer composite in the form of fatigue performance, strength, and specific stiffness. However, the interfacial and compression properties of the fiber reinforced polymer composite are dependent on the combination of characteristics of the interface between the fiber and the matrix, and combination of each primary component (Veedu et al., 2006).

For a continuous fiber-reinforced composite, the interface between the fiber and matrix is critical as it acts as a bridge that transfers load between the fiber and the matrix through shear flow, therefore, it is challenging for composites with high interfacial shear strength to transfer stress for reinforcement purposes (Lv et al., 2011). However, advancement of technology has introduced a hierarchical structures to improve the interfacial strength of fiber composite (Thostenson et al., 2002). Among several nano materials available, carbon nanotubes has been introduced into conventional fiber composite due to their remarkable thermal and electrical property, thermal property, outstanding modulus and strength, and unique structure as reported previously (M. Li et al., 2013).

There are two methods to incorporate nanofillers which are direct dispersion of nanofillers into composite matrix and direct attachment of nanofillers onto carbon fiber surface (M. Li et al., 2013). The techniques are direct mixing, electrophoretic deposition, chemical vapor deposition, and dipping deposition. Researches has paid attentions to directly attach carbon nanotubes onto carbon fiber due to convenience of fabrication of composite using traditional methods (M. Li et al., 2013). Previous study showed that fabrication of fiber polymer composite may introduce flaws and defects (Koushyar et al., 2012). The most critical defect is void formation and therefore, few strategies and approaches have been studies to reduce the defect.

### 2.1.2 Methods of Incorporating Fillers onto Carbon Fiber

The incorporation of fillers onto the carbon fiber is to enhance interfacial bonding between the fiber and matrix (L. G. Tang & Kardos, 1997). Mechanical properties of CFRP are highly dependent on the interaction and adhesion between fiber and the matrix (Chukov et al., 2014). The researcher reported that CF are chemically stable and smooth therefore exhibit low adhesion to the matrix. There are many methods of incorporating fillers onto carbon fibers which includes in-situ growth deposition (EPD and CVD), dipping deposition (DD), and direct mixing. The advantage and disadvantages can be referred in Table 2.1.

Table 2.1 Summary of deposition methods

| Methods | Advantages | Disadvantages | References |
|---|---|---|---|
| Electrophoretic Deposition (EPD) | • Inexpensive<br>• Produce uniform particulate with high green densities.<br>• Controlled thickness. | • Hydrogen evolution produces bubbles that can reduce mechanical properties. | (Basu et al., 2001)<br>(Thomas et al., 2005) |
| Chemical Vapor Deposition (CVD) | • Versatile technique | • Low deposition rate<br>• Expensive fabrication cost | (Bonard, 2006)<br>(Kim & Park, 2005) |

| | | | |
|---|---|---|---|
| | • Can grow wide range of carbon structure.<br>• Properties of structure can be controlled over a large range. | when mass produced. | |
| Dipping deposition | • Facile<br>• Economic<br>• Good machinability<br>• Widespread application | • Poor uniformity<br>• Relative weak bonding conditions | (X. Tang & Yan, 2017)<br><br>(Qi et al., 2014) |
| Direct Mixing | • Efficient way to disperse nanotubes with solvent aided dispersion.<br>• Does not require pre-dispersion stage. | • Agglomeration take place before epoxy can fully cure<br>• Produces lowest desired mechanical properties. | (Martone et al., 2012)<br><br>(Yasmin et al., 2006) |

2.1.2.1 Electrophoretic Deposition (EPD)

EPD is a type of colloidal process that has possess the benefit of short deposition time, able to deposit on complex object and requires simple apparatus (Besra & Liu, 2007). A number of researches has succeeded in performing EPD in various types of substrate and suspensions. Moreover, for CNT to be combined with other materials, it is important for it to be dispersed homogeneously in appropriate suspension (Boccaccini et al., 2006). Theoretically, electrophoresis are particles that are in motion due to Coulombic forces with nonzero net charge (Francis Avilés et al., 2018). Particles are observed and separated by characterizing their surface charge that are finite and particles movement in uniform field (Morgan & Green, 2003). Avilés et al. (2018), added that Coulomb force (Fc) of a particle is the result of product of the charge on the particle and electric field €. This relation is conveyed by the equation;

$$F_c = \int_S \sigma_q E \, dS \qquad \text{Equation 2.1}$$