Faculty of Manufacturing Engineering

PREPARATION AND CHARACTERISATION OF EPOXY/KENAF/MWCNTs HYBRID COMPOSITES

Noraina Alia binti Mat Noor

Master of Science in Manufacturing Engineering

2019

PREPARATION AND CHARACTERISATION OF EPOXY/KENAF/MWCNTs HYBRID COMPOSITES

NORAINA ALIA BINTI MAT NOOR

A thesis submitted
in fulfillment of the requirements for the degree of Master of Science
in Manufacturing Engineering

Faculty of Manufacturing Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2019

DECLARATION

I declare that this thesis entitled "Preparation and Characterisation of Epoxy/Kenaf/MWCNTs Hybrid Composites" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : .....

Name : .....

Date : .....

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering.

Signature :…….....

Supervisor Name :…….....

Date :…….....

DEDICATION

Special gift for my beloved father and mother who always trust and see the best in their daughter's well being. There is no other greater loves I can receive except from you, that always show strong support for me to fulfil my dream. Special appreciation to my beloved brothers and sisters: Syafiq, Syakir, Kak Anis, Adik and our late Baby Iman who shows the fullest encouragement and share undivided understanding for me to achieve my dream. To my respective supervisor and lecturers, thank you for the lesson and knowledge. To all my friends and every person involved, your support is very well appreciated. Sometimes dreams really come true.

ABSTRACT

This research is to prepare and characterize epoxy/kenaf single-composites and MWCNTs filled epoxy/kenaf multi-scale hybrid composites for the resulted mechanical, thermal, physical and morphological properties. The composites are prepared by using a high speed mechanical stirring process with curing by the solution casting technique. At the first phase of this research, the parameter interaction study and optimization between kenaf fibre content (-5.00 wt. %; +35.00 wt. %), stirring time (-10.00 minutes; +50.00 minutes) and stirring speed (-100 rpm; +700 rpm) for epoxy/kenaf composite system was established by using two-level full factorial design. A set of 2^3 fractional factorial design for three independent variables, with three replication at centre point and no blocks was used to yield a total of eleven (11) sets of experiments. The Design Expert 6.0.8 software was used to optimize the stirring parameters and kenaf filler loading towards the maximum tensile strength (TS) response. The optimum stirring conditions was optimized into stirring speed at 100 rpm, stirring time at 10 minutes and 5 wt. % of kenaf filler content, with the highest repeatabilityR^2 value of ~99.9%. The stage two of this research had specifically focus on the effect of MWCNTs nanofiller weight percentages addition into the epoxy/kenaf composites at 0.00, 0.25, 0.50, 0.75, 1.00 and 3.00 wt. %.The addition of MWCNTs as second reinforcement phase in epoxy/kenaf composites could enhanced the interfacial adhesion and provided good synergistic effects toward the properties enhancement for the epoxy/kenaf/MWCNTs hybrid composites. The multi-scale hybrid composites with 1.00 wt. % of MWCNTs addition, shows outstanding mechanical properties improvement (tensile strength: +48.24% and tensile modulus: +44.59%) as compared than epoxy/kenaf composite (controlled sample).The experimental results were supported with thermal and dynamic analyses. Addition of 1.00 wt.% MWCNTs into epoxy/kenaf composite have shifted glass transition temperature (T_g) at 43.90°C from 41.90°C for epoxy/kenaf composite without MWCNTs. This indicates the role of MWCNTs in improving thermal stability of the hybrid composite. In overall, MWCNTs filled epoxy/kenaf multi-scale hybrid composite, prepared by using an optimized stirring conditions and kenaf fibre loadings was able to provide hybrid synergism between both fillers and the epoxy matrix. Hence, contributed to significant improvement in hybrid composite stiffening effect.

ABSTRAK

Kajian ini dijalankan untuk menyediakan dan mencirikan komposit tunggal epoksi/kenaf dan komposit hibrid MWCNTs isian epoksi/kenaf terhadap prestasi mekanikal, terma, fizikal dan morfologi. Komposit ini disediakan dengan menggunakan proses pengadukan berkelajuan tinggi dengan pematangan secara teknik tuangan larutan. Pada fasa pertama penyelidikan, kajian interaksi parameter dan pengoptimuman antara kandungan gentian kenaf (-5.00 wt. %; +35.00 wt. %), masa pengadukan (-10.00 minit; +50.00 minit) dan halaju pengadukan (-100 ppm; +700 ppm) bagi sistem komposit epoksi/kenaf telah disahkan dengan menggunakan reka bentuk faktorial penuh dua-peringkat. Satu set 2^3 reka bentuk pecahan faktorial dengan tiga pembolehubah bebas, tiga replikasi pada titik tengah dan tiada blok telah digunakan bagi menghasilkan sebelas (11) set ekperimen. Perisian Design Expert 6.0.8 telah digunakan untuk mengoptimumkan parameter pengadukan dan penambahan gentian kenaf ke atas respon maksimum bagi kekuatan tegangan (TS). Keadaan pengoptimuman pengadukan telah dioptimumkan kepada kelajuan pengaduk pada 100 ppm, 10 minit tempoh pengadukan dan 5 wt. % kandungan gentian kenaf, dengan kebolehulangan yang tinggi, R^2sebanyak ~99.9%. Peringkat kedua kajian memfokus secara spesifik terhadap kesan penambahan peratus berat pengisi nano-MWCNTs terhadap komposit epoksi/kenaf pada 0.00, 0.25, 0.50, 0.75, 1.00 dan 3.00 wt. %. Penambahan MWCNTs sebagai fasa penguat kedua pada komposit epoksi/kenaf telah meningkatkan lekatan antara muka dan memberikan kesan baik sinergistik terhadap peningkatan sifat komposit hibrid epoksi/kenaf/MWCNTs. Komposit hibrid pelbagai skala dengan penambahan 1.00wt.% MWCNTs menunjukkan perubahan sifat mekanikal terbaik (kekuatan tegangan: +48.24% dan modulus tegangan: +44.59%) berbanding dengan komposit epoksi/kenaf (sampel kawalan). Keputusan ujikaji telah disokong oleh analisis haba dan dinamik. Penambahan 1.00 wt.% MWCNTs kepada komposit epoksi/kenaf telah menganjak suhu peralihan kekaca (T_g) kepada 43.90°C daripada 41.90°C, bagi komposit epoksi/kenaf tanpa MWCNTs. Ini menunjukkan peranan MWCNTs dalam memperbaiki kestabilan haba komposit hibrid. Secara keseluruhan komposit hibrid pelbagai skala epoksi/kenaf berpengisi MWCNTs yang disediakan dalam keadaan pengadukan dan penambahan gentian kenaf yang optimum, dapat memberikan kesan sinergi antara kedua-dua matrik epoksi dan pengisi, seterusnya menyumbang kepada kesan penguatan yang signifikan.

ACKNOWLEDGEMENTS

In the name of Allah, the most gracious, the most merciful, source of never ending knowledge, I am very grateful with all the patience, blessing and good health that has been given to me till this research came to realization.

I would like to express the highest gratitude and appreciation to my respected supervisor Dr. Jeefferie bin Abd Razak for his supervision, never ending support, advice, information, and guidance toward the completion of this research. I also would like to show my special thanks to my co-supervisor, Dr. Syahriza binti Ismail for her assistance, knowledge and guidance in my study period.

Next, I would like to thank all staff of Faculty of Manufacturing Engineering and staff of Centre for Graduate Studies, Universiti Teknikal Malaysia Melaka (UTeM) for providing facilities, supports and great cooperation. I am particularly thankful to UTeM and Ministry of Higher Education, Malaysia (MOHE) for the financial support through the Research Acculturation Grant Scheme (RAGS/1/2014/TK04/FKP/B00072).

I would like to thank my family: my parents and to my brothers and sisters for supporting me spiritually throughout my thesis writing and during the candidature period.

Finally, my special thanks also for my beloved family members and all my friends who always provides continues support and never-ending motivation to me.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS AND SYMBOLS

ANOVA	–	Analysis of Variance
ASTM	–	American Standard Testing of Materials
CNTs	–	Carbon Nanotubes
DMA	–	Dynamic Mechanical Analysis
DOE	–	Design of Experiment
DSC	–	Differential Scanning Calorimeters
EAB	–	Elongation at Break
HRTEM	–	High-Resolution Transmission Electron Microscope
MWCNTs	–	Multi-walled CNTs
RSM	–	Response Surface Methodology
SEM	–	Scanning Electron Microscope
SWCNTs	–	Single-walled CNTs
TEM	–	Transmission Electron Microscope
VPSEM	–	Variables Pressure Type of SEM
℃	–	Degree Celsius
E’	–	Storage Modulus
E”	–	Loss Modulus
tan δ	–	tan delta
%	–	Percent

LIST OF PUBLICATIONS

Noor, N.A.M., Razak, J.A., Ismail, S., Mohamad, N., Tee, L. K., Munawar, R.F., Junid, R., 2017. Review on Carbon Nanotube based Polymer Composites and Its Applications. *Journal of Advanced Manufacturing Technology*, pp. 311 – 326.

Noor, N.A.M., Razak, J.A., Ismail, S., Mohamad, N., Yaakob, M.Y., Theng, T.H., 2017. Influence of MWCNTs Addition on Mechanical and Thermal Behaviour of Epoxy/Kenaf Multi-scale Nanocomposite. *IOP Conference Series: Materials Science and Engineering*, 210 (012058).

Noor, N.A.M., Razak, J.A., Ismail, S., Mohamad, N., Junid, R., 2017. MWCNTs Functionalization and their Effects on the Mechanical Properties of Epoxy/MWCNTs Nanocomposites. *Proceedings of Innovative Research and Industrial Dialogue'16,* pp. 93 – 94.

Noor, N.A.M., Razak, J.A., Ismail, S., Mohamad, N., Othman, I.S., Theng, T.H., Yaakob, M.Y., 2017. Stiffness and Fracture Toughness Behaviour of Kenaf Fiber reinforced MWCNTs/Epoxy Multi-scale Composites. *Proceedings of Mechanical Engineering Research Day*, pp. 348 – 350.

Noor, N.A.M., Razak, J.A., Ismail, S., Mohamad, N., Othman, I.S., Theng, T.H., Yaakob, M.Y., Full-length Manuscript Accepted. Stiffness and Fracture Toughness Behaviour of Kenaf Fibre reinforced MWCNTs/Epoxy Multi-scale Composites. *Journal of Mechanical Engineering (JMECHE).*

LIST OF ACHIEVEMENTS AND INTELECTUAL PROPERTY

1. Registered one copyright, Title of copyright: Eco-Door Automotive Inner Panel, Notification Number: CRLY00008204
2. Silver Medalist in 28th ITEX 2017, Product Name: Multi-scale Eco-composite Automotive Door Panel
3. Silver Medalist in PECIPTA 2017, Project Title: Nano-Eco Door
4. Gold Medalist in Mini UTEMEX 2016, Project title: Kenaf-MWCNTs Multi-scale Eco-Composite Automotive Door Panel
5. Gold Medalist in 14th Perkaya Inovasi PMM, Project title: MWCNTs Functionalization and their Effect on the Mechanical Properties of Epoxy Nanocomposite

CHAPTER 1

INTRODUCTION

1.1 Background of study

Composite is a wonder material that plays an essential part in the development of mankind history. Composite material is well-known to offer the advantages such as light-weight, corrosion resistance and high fatigue strength. They are extensively used as materials in making of aircraft structures, electronic packaging to medical equipment, and space vehicle to home building (Shaw et al., 2010). Composite can be categorized into three main important group which are ceramic matrix composites (CMC), metal matrix composites (MMC) and polymer matrix composite (PMC). In this research, we are focusing on polymer matrix composite containing multi-scale hybrid filler system, which consisted of kenaf fibre and multi-walled carbon nanotubes (MWCNTs) within an epoxy matrix.

Carbon nanotubes (CNTs) have becoming as one of the most influential reinforcing elements for polymeric based composite materials since its discovery in 1991 by Iijima (Siddique and Mehta, 2014). It is characterized to possess high strength and modulus along with higher temperature resistance. This carbonaceous nanofillers offer a very promising role in respect to their better structural and functional properties (Ma et al., 2010). Due to these outstanding mechanical properties as well as higher aspect ratio and lower density, CNTs can be ideally used as candidates for polymer composite reinforcement applications (Karimi et al., 2017). CNTs are normally mixtures of various chirality, diameters, and lengths. Furthermore, CNT aggregation has been found to dramatically hamper the