



**Experimental and Numerical Investigation of
Hydrothermal Effect on Mechanical Properties of
Adhesively Bonded T-Joint**

by

Siti Nurhashima Binti Mohd Isa

1431411203

A thesis submitted in fulfilment of the requirements for the degree of
Master of Science in Mechanical Engineering

**School of Mechatronics Engineering
UNIVERSITI MALAYSIA PERLIS**

2016

ACKNOWLEDGEMENT

In the name of Allah, The Most Gracious and Most Merciful. First of all, I thank Allah for giving me the patience, strength and effort to complete this study.

I would like to express my gratefully thanks to my supervisor, Dr. Mohd Afendi Bin Rojan for his support throughout the process of this research. With his advice, guidance and willingness of sharing the knowledge help in every aspect of the work, all difficulties and problems that I faced. To my co-supervisor, Dr Nasrul Amri Bin Mohd Amin, my sincere gratitude for his advice and his assistance towards the process of completing my work successfully.

Special thanks dedicated to Petronas Chemical Fertilizer Kedah (PCFK) Sdn. Bhd for providing the equipment and sharing the information which made this research at ease, and also to Universiti Malaysia Perlis (UniMAP), which is greatly acknowledged for giving me the platform to further my studies and gaining more information.

Besides that, I would like to thank the staff from the laboratory, especially Mr. Muhammad Alif Bin Mad Yussof, for the meaningful assistance in conducting the tests, and the personnel of the Adhesives Group for their accessibility to help and support me in any situation that arose throughout the process.

Finally, I would like to express my token of appreciation to all my friends and family who have been supportive in every stage of my master's degree throughout the years making the progress of this thesis a pleasant and all around enjoyable experience. It was their care and understanding that enabled the accomplishment of this important step of my life.

TABLE OF CONTENTS

	PAGE
THESIS DECLARATION	i
ACKNOWLEDGEMENT	ii
TABLE OF CONTENTS	iii
LIST OF FIGURES	vii
LIST OF TABLES	xi
LIST OF ABBREVIATIONS	xii
LIST OF SYMBOLS	xiii
ABSTRAK	xiv
ABSTRACT	xv
CHAPTER 1 INTRODUCTION	
1.1 Background	1
1.1.1 Urea granulator fluidization bed	3
1.2 Problem Statement	5
1.3 Research objectives	5
1.4 Research scope	6
1.5 Thesis outline	7
CHAPTER 2 LITERATURE REVIEW	
2.1 Introduction	9
2.2 Adhesive joint	9

2.3 Hydrothermal effect on adhesive	12
2.4 Diffusion in epoxy adhesive	16
2.4.1 Fickian diffusion	19
2.4.2 Non-Fickian Diffusion	20
2.5 Diffusion in adhesive joints	21
2.6 Modelling adhesive joints by finite element	23
2.7 Chapter summary	26
CHAPTER 3 RESEARCH METHODOLOGY	
3.1 Introduction	27
3.2 Materials and processing	30
3.2.1 Materials	30
3.2.1 Processing	32
3.3 Environmental condition	35
3.4 Testing method	35
3.4.1 Water absorption test	35
3.4.2 Calorimetric analysis	38
3.4.3 Thermo gravimetric Analysis	39
3.4.4 Compression test of bulk adhesive	40
3.4.5 Tensile test of T-joint	41
3.5 Scanning electron microscope	42
3.6 Finite element analysis	43
3.6.1 Material properties	43

3.6.2 Material model	44
3.6.3 Geometry and meshing	45
3.6.4 Loading and boundary condition	46
3.6.5 Effect of boundary condition	48
3.7 Chapter summary	52
CHAPTER 4 RESULTS AND DISCUSSION	
4.1 Introduction	53
4.2 Water absorption	53
4.2.1 Moisture uptake	53
4.2.2 Fickian and non-Fickian behaviour	56
4.2.3 Diffusion parameter	58
4.3 Thermal characteristic	59
4.3.1 Differential Scanning Calorimetric	59
4.3.2 Thermo Gravimetric Analysis	61
4.4 Morphology of epoxy adhesive surface	63
4.5 Mechanical properties	66
4.5.1 Compression test of bulk specimen	66
4.5.2 Tensile test of T-joint specimen	69
4.6 Failure mode of T-joint specimen	72
4.7 Finite element validation with experiment	73
4.7.1 Linearised maximum stress	73

4.7.2 Stress distribution	76
4.7.3 Failure load prediction of adhesive joint	79
4.8 Chapter summary	82
CHAPTER 5 CONCLUSION AND FUTURE WORK	
5.1 Introduction	83
5.2 Conclusion	83
5.3 Contribution to knowledge	85
5.4 Future work	86
REFERENCES	87
APPENDIX A	96
LIST OF PUBLICATIONS	99

©This item is protected by original copyright

LIST OF FIGURES

NO.	PAGE
1.1: China adhesives market volume by technology, 2012-2020 (Kilo Tons).	2
1.2: (a) Schematic diagram of urea granulator system, (b) actual urea granulator fluidization bed.	4
1.3: Perforated plate lift off due to joint failure.	4
2.1: Structural of adhesive joint (Rosendo, 2015).	10
2.2: Joint configuration (He, 2011).	11
2.3: (a) Electron cloud distribution and (b) hydrogen bonding of water molecule (Ferguson & Qu, 2007).	18
2.4: Hydrophobic and hydrophilic water contact angle behaviour (Ferguson & Qu, 2007).	18
2.5: Illustration of Fickian diffusion (Wong., 2013).	19
2.6: Illustration of non-Fickian diffusion (Wong, 2013).	21
3.1: Flow chart of research methodology.	28
3.2: T-joint configuration (a) T-joint specimen, (b) Stainless steel plate, (c) Stainless steel perforated plate.	30
3.3: (a) Stainless steel perforated plate, (b) Stainless steel plate and (c) Teflon adjuster.	31
3.4: Araldite epoxy adhesive.	31
3.5: Colour of Araldite epoxy adhesive after mixing.	32

3.6: (a) Teflon mould of bulk adhesive and (b) Dimension of bulk adhesive.	33
3.7: (a) Teflon adjuster, (b) T-joint preparation.	34
3.8: Determination of glass transition temperature, T_g .	39
3.9: (a)-(e) Compression test and (f) specimen after compression test.	40
3.10: T-joint under tensile loading.	41
3.11: Platinum coating.	42
3.12: TM3000 Scanning electron microscope (SEM).	42
3.13: True stress-strain for RTD and EWT.	45
3.14: Finite element mesh.	46
3.15: Loading and boundary conditions under tensile loading (a) and perforated plate constrains at (b) four side, (c) two side and (d) eight vertex.	47
3.16: Failure force against thickness for different boundary condition.	48
3.17: Deformation of perforated plate.	50
3.18: Contour deformation of perforated plate constraint (a) four side, (b) two side and (c) eight vertexes.	51
4.1: Moisture uptake against time for EWT.	55
4.2: Fitted curve to check the coefficient of correlation, n.	56
4.3: Fickian fit curve of EWT.	57
4.4: Arrhenius relationship to determine activation energy.	59
4.5: DSC curve of RTD and EWT.	60

4.6: TGA curve of RTD and EWT.	62
4.7: DTA curve for RTD and EWT.	62
4.8: Morphology surface structure (a) RTD and EWT (b) 80°C, (c) 90°C and (d) 100°C.	65
4.9: Compressive strength against immersion time for EWT.	67
4.10: Stress-strain curve for RTD and EWT.	69
4.11: Failure load against adhesive bond thickness (a) RTD and EWT (b) 80°C, (c) 90°C and (d) 100°C.	70
4.12: Average failure load against bond thickness for RTD and EWT.	72
4.13: Failure of T-joint specimen at bond thicknesses (a) 0.5mm, (b)1.0mm, (c) 1.5mm and (d) 2.0mm.	73
4.14: Linearised maximum stress in horizontal and vertical paths of adhesive.	74
4.15: Linearised maximum stress for RTD and EWT in horizontal path at bond thicknesses (a) 0.5mm, (b) 1.0mm, (c) 1.5mm and (d) 2.0mm.	75
4.16: Linearised maximum stress in vertical path for RTD and EWT at bond thickness (a) 0.5mm, (b) 1.0mm, (c) 1.5mm and (d) 2.0mm.	76
4.17: Stress distribution in adhesive for RTD condition at bond thickness (a) 0.5mm, (b) 1.0mm, (c) 1.5mm and (d) 2.0mm.	77
4.18: Stress distribution in adhesive layer for (a) RTD and EWT (b) 80°C, (c) 90°C, (d) 100°C.	78

4.19: Yield stress of bulk adhesive and Von Mises stress of T-joint for RTD and EWT at different bond thicknesses. 80

4.20: Failure load against bond thickness (a) RTD and EWT (b) 80°C, (c) 90°C and (d) 100°C. 81

©This item is protected by original copyright

LIST OF TABLES

NO.	PAGE
2.1: Types of transport and diffusion exponent (n)	16
3.1: Mechanical properties of adhesive-adherent	44
4.1: Justification of Fickian behaviour	57
4.2: Values of diffusion coefficient, equilibrium moisture, permeability and crosslink density	58
4.3: Decomposition stages loss, peak degradation temperature and percentage of weight loss for RTD and EWT at 80°C, 90°C and 100°C	63
4.4: Value of compressive strength and elastic modulus for conditions tested	67

©This item is protected by original copyright

LIST OF ABBREVIATIONS

DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
EWT	Elevated Water Temperature
FE	Finite Element
FEA	Finite Element Analysis
MISO	Multi-linear Isotropic Hardening
RTD	Room Temperature and Dry
SEM	Scanning Electron Microscope
TGA	Thermogravimetric Analysis
UTM	Universal Tensile Machine
VOC	Volatile Organic Compound

©This item is protected by original copyright

LIST OF SYMBOLS

C	Water concentration
D	Diffusion coefficient
D_0	pre-exponential coefficient
E	Activation energy
E_0	Young's modulus
F	Failure force
F'	Prediction of failure force
h	Thickness
M_0	Mass of dry specimen
M_s	Equilibrium moisture uptake
M_t	Moisture uptake at time
n	Coefficient of correlation
N	Number of crosslink
P	Permeability
t	Time
R	Gas constant
T	temperature
T_g	Glass transition temperature
σ	Yield stress of bulk adhesive
σ'	Von Mises stress

Penyiasatan Ekperimen dan Berangka Terhadap Kesan Hidroterma Pada Ciri-ciri

Mekanikal Ikatan Perekat Sendi-T

ABSTRAK

Persekitaran yang lembap adalah satu masalah yang penting dalam mereka bentuk perekat dalam apa-apa aplikasi. Dasar pembendaliran urea telah dibasuh dengan air panas oleh itu mencipta faktor persekitaran yang lembap. Oleh itu, keadaan ini dirujuk sebagai satu masalah dalam mereka bentuk perekat sendi-T untuk dasar pembendaliran urea. Tujuan kajian ini adalah untuk mengkaji kesan hidroterma perekat sendi-T dengan perekat berbeza ketebalan dalam ujian air panas pada suhu 80°C, 90°C dan 100°C. Dua keadaan persekitaran yang dikaji, suhu bilik dan kering (RTD) dan suhu air yang ternaik (EWT) pada 80°C, 90°C dan 100°C direndam selama 15 minit. Beberapa ketebalan ikatan yang dikenal pasti untuk dikaji adalah 0.5mm, 1.0mm, 1.5mm dan 2.0mm. Tambahan lagi, kekuatan ikatan yang bergantung kepada kelembapan dinilai dengan membandingkan ciri-ciri berikut dengan nilai pada suhu bilik. Bebanan unipaksi dilakukan menggunakan ujian mampatan untuk specimen pada keadaan RTD dan EWT. Eksperimen yang melibatkan specimen sendi-T dengan bebanan tegangan untuk perekat yang berbeza ketebalan telah dijalankan. Akhir sekali, prestasi aplikasi sendi-T dalam eksperimen dibandingkan dengan model geometri sendi-T dalam perisian menggunakan analisis unsur terhingga (FEA) ANSYS 14.0. Tegasan kegagalan ditentukan sebagai kriteria untuk menyiasat prestasi perekat. Hasil kajian dikemukakan dengan ketebalan perekat yang terbaik dan persekitaran kelembapan untuk perekat epoksi Araldite. Kehadiran lembapan secara langsung pada permukaan perekat mengubah integriti antaramuka sendi perekat. Perekat epoksi mempunyai kesan ketara dalam kekuatan ikatan antaramuka selepas direndam dalam air panas. Walau bagaimanapun kekuatan ujian specimen sendi-T yang direndam dalam air panas pada suhu 80°C dan ketebalan ikatan 1.5mm mempunyai kekuatan tinggi dengan specimen sendi-T pada RTD. Selain itu, kekuatan ujian mampatan juga menunjukkan perilaku pengurangan kekuatan yang sama apabila perekat direndam dalam air panas. Seterusnya, pendekatan untuk meramalkan hasil eksperimen menggunakan perisian unsur terhingga yang dikomersilkan, ANSYS 14.0 memberi persetujuan yang baik dengan corak lengkung sama dengan lengkung kegagalan tegasan. Oleh itu, model simulasi yang dikaji dan diramalkan boleh digunakan untuk mensimulasikan perekat sendi-T untuk pelbagai keadaan sempadan.

Experiment and Numerical Investigation of Hydrothermal Effect on Mechanical Properties of Adhesively Bonded T-joint

ABSTRACT

The moisture environment is a significant problem in designing the adhesive joint in any application. Urea fluidisation bed was washed with hot condense water thus created moisture environmental factor. This situation was cited as a problem in designing adhesively bonded T-joint referring joint part in urea fluidization bed. The purpose of this study was to examine hydrothermal effect on adhesively bonded T- joints with different adhesive thickness in hot water test at temperatures of 80°C, 90°C and 100°C. Two environmental conditions were studied, namely room temperature and dry (RTD) and elevated water temperature (EWT) at 80°C, 90°C and 100°C immersed for 15 minutes. Various bond thickness involved in testing namely 0.5mm, 1.0mm, 1.5mm and 2.0mm. Moreover, the moisture dependence of joint strength was evaluated by comparing those properties with the values at room temperature. Uniaxial loading was performed using a compression test of bulk specimen for both RTD and EWT condition. Another series of tests was run involving T-joint specimen with tensile loading for different adhesive thickness. Finally, the performance of the T-joint application in experiment was compared with the geometrical modelling of T-joint in ANSYS 14.0 software finite element analysis (FEA). Moreover, failure stress was determined as a criteria to investigate the adhesive performance. Results were presented for the best adhesive thickness and moisture environment for Araldite epoxy adhesive. Direct presence of moisture at the adhesive interface alters the interfacial integrity of the adhesive joint. However, the strength of test T-joint specimen immersed in 80°C of hot water and bond thickness 1.5mm appeared to have high strength compared with T-joint specimen at RTD. Moreover, the compressive strength also showed similar behaviour of reductions under the hot water condition. Furthermore, the approach to predict an experimental result using the commercialised finite element software, the ANSYS 14.0 resulted in a good agreement of similar pattern of failure stress curves. The simulation model has been predicted, thus can be used to simulate the T-joint and adhesives at numerous boundary conditions.

CHAPTER 1

INTRODUCTION

1.1 Background

An adhesive is a substance that is capable of holding materials together by the surface attachment. The use of structural adhesives in the industry has grown extensively in recent years. This can be attributed to a number of desirable qualities which adhesive bonding allows in comparison with more traditional joining techniques such as riveting and welding. In addition, some of the advantages that the use of adhesives can offer are the ability to join dissimilar materials efficiently, being the most convenient and cost effective technique and moreover, having the adhesive increase flexibility in bonding design (Baldan, 2004).

According to Grand View Research Market Research and Consulting (2014), China's adhesive market shows an increasing volume from 2012 to 2020 in respect to global classified adhesive, water-based, solvent-based, hot melt, and reactive on the basis of technology as shown in Fig. 1.1. Adhesive is an eco-friendly nature with zero volatile organic compound (VOC) emission contributing the main factor to a higher demand.

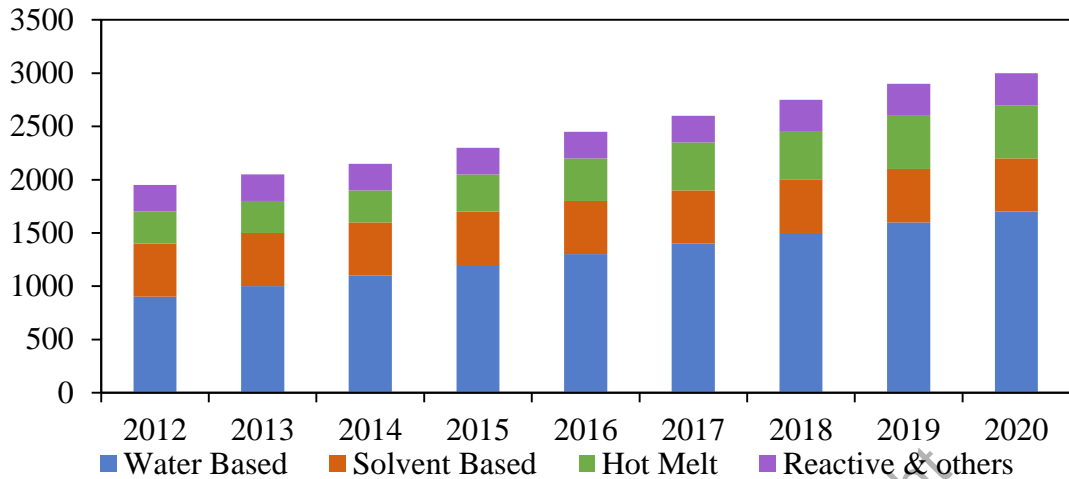


Figure 1.1: China adhesives market volume by technology, 2012-2020 (Kilo Tons).

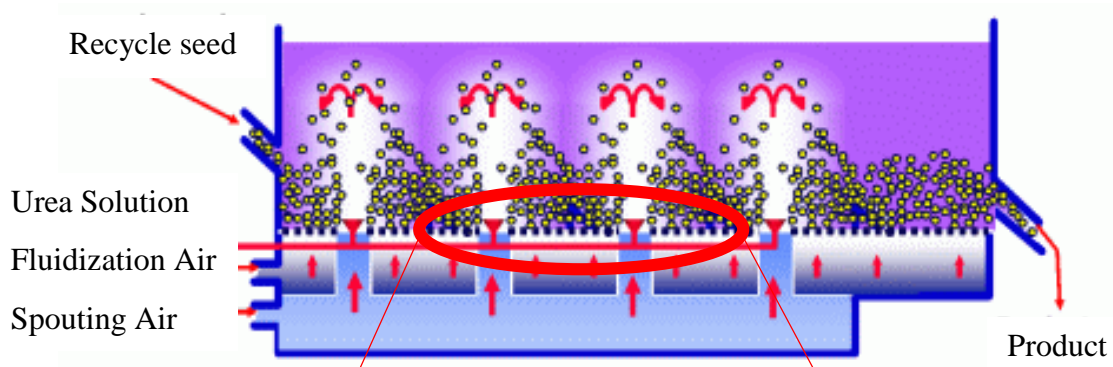
Many considerations need to be taken into account while choosing the compatible adhesive. Environmental effect on the adhesive is an important attention and aspect in the early stage of design. Water is the hostile agent in degrading the performance of adhesive joint while the durability of the adhesive joint reduces after long exposure in the water (Mubashar, Ashcroft, Critchlow, & Crocombe, 2009). Furthermore, modulus, strength and strain are also affected by the moisture environment as compared to specimen in a normal environment (Abdel-Magid, Ziaee, Gass, & Schneider, 2005). This limitation addresses one of the most fundamental problems in an attempt to make the adhesive a bonding application in the industry.

The transport and movement of water molecule in the adhesive structure are critical investigation aspect in order to study the adhesive degradation behaviour. The effects of moisture to the adhesive applications are difficult to avoid because water naturally exists in the normal atmosphere environment. Besides that, moisture absorption usually degrades adhesive performance, strength and reduce the glass transition temperature, T_g . Therefore, the effects of moisture content in adhesive are very significant criteria in designing an adhesive application (Li, 2000).

1.1.1 Urea granulator fluidization bed

Granulator fluidization bed is a main component of the urea granulator system. By utilising thin stainless steel perforated plate, fluidization air from blower passes through the perforated plate to form air cushion. Air cushion is very crucial for newly formed granules to travel out from the granulator. It is noted that suitable amount of water is important during granulation process (Miwa, Yajima, Ikuta, & Makado, 2008). The schematic diagram urea granulator fluidization bed from Toyo engineering Cooperation was shown in Fig.1.2 (a) and the actual urea granulator fluidization bed in Fig. 1.2 (b).

Spot welding is used to join perforated plate to its frame structure. This technique possesses unreliable joining due to high stress concentration and vibration of the component (Deshmukh, Burande, Shukla, & Kamble, 2014). Joining failure causes granulator to immediately shutdown, which costs a significant loss to the company. Therefore in this research, uniaxial loading will be conducted by referring the force exist in the fluidization bed (Papiya Roy, Rajesh Khanna, 2008). Finite element analysis test under hydrothermal condition will be performed as a case viable to study adhesive bonding for urea granulator fluidisation bed. Fig. 1.3 shows perforated plate lift off due to joint failure.



(a)



(b)

Figure 1.2: (a) Schematic diagram of urea granulator system, (b) actual urea granulator fluidization bed.



Figure 1.3: Perforated plate lift off due to joint failure.

1.2 Problem Statement

Petronas Chemical Fertilizer Kedah (PCFK) Sdn. Bhd used spot weld to join the perforated plate in urea granulator fluidisation bed. However, very particular welding technique requirement causes the weakness to the plate materials properties and the thin plate lifted off during the granulation process. Thus adhesive was proposed as an option joint technique. Environment condition is essential consideration while design the adhesive joint. Urea fluidization bed involved wet cleaning routine with hot condense of water once a month.

The adhesive bonded joint is study in this research regarding hydrothermal effect for urea fluidization bed exposed in the humid environment. An appropriate joint strength and effective bond thickness of adhesive perhaps can avoid joint fail. This adhesive under hydrothermal effect should be investigated to provide joint strength properties as compared to normal room temperature. Finite element analysis also are required in order to validate with experimental investigation.

1.3 Research objectives

This research aims to extract the understanding on the adhesive bonding which requires further investigation on variation parameter of hydrothermal effect and thickness under uniaxial loading. The finding from this study can be used to develop the adhesively bonded T-joint. The objectives of this study are addressed as follows;

- i. To examine the hydrothermal effect of moisture to adhesive joint strength behaviour specimen exposed in two conditions; room temperature and dry (RTD) and elevated water temperature (EWT).

- ii. To evaluate the influence of different adhesive bond thicknesses by comparing those properties with the values at RTD and EWT.
- iii. To predict the failure strength of adhesively bonded T-joint subjected to tensile loading with the values at RTD and EWT using finite element model and compared with the experimental result.

1.4 Research scope

In this study, the hydrothermal effect on epoxy adhesive with variable bond thickness is investigated in parametric studies. The material in this study involves stainless steel 304 containing two parts, namely stainless steel plate with dimension of 100mm X 100mm X 10mm and stainless steel perforated plate with dimension of 100mm X 80mm X 1.25mm. These specimens are made from actual 1.25mm perforated plate to simulate the actual structure of fluidisation bed in PCFK granulator equipment. Araldite epoxy adhesive was used in this study.

The specimen is exposed into two conditions, room temperature and dry (RTD) and elevated water temperature (EWT) for bulk adhesive specimen and T-joint specimen. Besides that, the experiments are conducted in the room temperature for both bulk adhesive and T-joint specimens subjected to axial loading; compression and tensile. The thickness of adhesive and temperature of water are considered as parameters in this investigation. The thickness was varied as 0.5mm, 1.0mm, 1.5mm and 2.0mm, while aging condition at water temperatures of 80°C, 90°C and 100°C were used in this study.

Other than that, the geometrical T-joint specimen is modelled in the Solid Work software and imported into ANSYS 14.0 for finite element analysis. The prediction of

finite element analysis (FEA) is studied in terms of failure stress at the adhesive part as the determination of failure criteria.

The effective thickness of adhesive and hot water is determined in this research by observing the performance of the adhesive under the moisture environment. Furthermore, the influence of water absorption on the deformation of the adhesive is investigated. The performance of the adhesive is estimated to be validated based on the failure stress by comparing the result from FEA and experiment works.

In general, this research can be separated into three main phases:

- Experimental works under compression and tensile loading.
- Parametric study with respect to variation thickness of the adhesive and different conditional exposes.
- Finite element analysis (FEA) development.

1.5 Thesis outline

Chapter 2 accesses a literature review of the research. Consideration in applying the knowledge to study the effect of water towards adhesive is reviewed from other researchers. Also, the environmental factors such as moisture and the adhesive joint designs observed from others studies are discussed, followed by the deviations of epoxy adhesive in terms of material properties and the chemical reaction when exposed into water.

Chapter 3 discusses the detail method used in the study, while materials and process to prepare the specimen are explained. Also, the environmental condition applied in the case study and the experimental procedure of the test conducted are presented

properly in this chapter. American Society for Testing and Material (ASTM) is referred as guidance to conduct the test and all procedures used in ANSYS software to predict and compare the result with experiment in terms of geometry, material declaration and loading applied are shows in this research methodology chapter.

Meanwhile, Chapter 4 explains the data obtained from the experiment and detail discussion of the result. Next, the test of water absorption by the adhesive is observed to see the behaviour of the absorption water by the Araldite epoxy adhesive. The strength of the adhesive is performed by compression and tensile loading is observed in the numerical data, followed by predicting the approach of the testing conducted by finite element analysis (FEA) in ANSYS. The physical effect to the adhesive surface due to water absorption is next viewed using Scanning Electron Microscope (SEM) and result of thermal effect is presented.

Chapter 5 summarises all the findings in this case study. The response of the adhesive when exposed in the moisture environment is concluded, and the contribution to knowledge of this study for future work is proposed.

CHAPTER 2

LITERATURE REVIEWS

2.1 Introduction

The literature review in this chapter focuses on the aspects which corresponds to the case study and related issues to the research objective. The information from other researchers is used as a guideline to structure the experiment and the analysis. Besides that, this literature review also helps to adequate all the steps and information required to represent the finding in proper order in which the environment exposed to the adhesive epoxy resin has been reviewed in other studies. The adaption of the adhesive in moisture condition by properties changes after the loading is presented in the literature review chapter.

2.2 Adhesive joint

An adhesive is a substance that is capable of holding materials together by the surface attachment, whereby materials being joined are called adherents. Adhesive has also acknowledged in the bonding structural application in many industries and technologies including automotive, aircraft and electronics. Besides that, many researchers have studied influential factors of adhesive bonding that cover a broad