EXPERIMENTAL AND FINITE ELEMENT STUDY OF EGGSHELL POWDER ON SHEAR STRENGTH OF ADHESIVELY BONDED JOINTS

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DEDICATION

This thesis is dedicated to my parents, who supported me during my studies by providing financial and mental support.

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ABSTRACT

Eggshells are daily food waste disposed of in landfills, producing environmental issues and an unpleasant odour. Eggshells were crushed to form eggshell powder and may be suitably applied as a filler in epoxy resins to improve their mechanical properties as neat epoxy resins have the drawbacks of low shear strength, low fracture toughness. In this study, dog-bone specimens were fabricated to investigate the mechanical properties of toughened epoxy with eggshell powder (TEEP), such as the elastic modulus, Poisson's ratio and tensile strength. Double Cantilever Beam (DCB) and End-Notched Flexure (ENF) tests were used to determine the Mode-I and Mode II fracture energy of TEEP specimens, respectively. For this purpose, oven-dried eggshells were crushed to obtain particles with a size of 150 μm. The volume fraction of eggshell powder in epoxy was designated as 0%, 2.5%, 5%, and 10%, respectively. The epoxy resin system was made by mixing EPIKOTE Resin 828 and Hardener 651 with a mixing ratio of 5:2 by weight. The results showed that the tensile strength, elastic modulus, Mode I and Mode II fracture energies of TEEP were optimum at 5% eggshell with the enhancement of 36.8%, 24.9%, 60.3% and 166.3%, respectively than those of neat epoxy. This is due to the ability of eggshell powder as a crack arrestor, indicated by rough fracture surfaces. However, 10% TEEP prone to agglomeration of eggshell powder lead to poor bonding with epoxy resin and substantially reduces mechanical properties. Subsequently, the shear strength of the single-lap joint (SLJ) with the 38.1mm overlap length, bonded with 5% TEEP has improved up to 72.7% compared to that of neat epoxy. The finite element modelling framework was developed using the Cohesive Zone Modelling (CZM) technique by incorporating the traction-separation relationship as a constitutive model. Here, the measured values were adopted, and good agreement with experimental datasets was found with average discrepancies less than 10.8%.

ABSTRAK

Kulit telur adalah bahan buangan harian yang biasanya dilupuskan di pusat perlupusan sampah yang menjurus kepada masalah alam sekitar dan bau yang tidak menyenangkan. Kulit telur dihancurkan menjadi serbuk kulit telur untuk dijadikan sebagai pengisi dalam epoksi resin untuk meningkatkan sifat mekanikalnya kerana epoksi resin biasa mempunyai kekuatan ricih yang rendah, dan tenaga patah yang rendah. Spesimen tulang anjing difabrikasi untuk mengkaji sifat mekanikal epoksi resin iaitu, (modulus elastik, nisbah Poisson dan kekuatan tegangan) dan diuji di bawah bebanan ujian tegangan. Ujikaji rasuk terjulur berganda (DCB) dan lenturan hujung takukan (ENF) digunakan untuk menentukan ujian patah spesimen TEEP. Untuk tujuan ini, kulit telur yang telah dikeringkan dihancurkan kepada saiz partikel 150µm. Nisbah separa serbuk kulit telur sebagai pengisi epoksi resin yang dikaji adalah masing-masing sebanyak 0%, 2.5%, 5%, and 10% relatif berat epoksi. Sistem epoksi resin dihasilkan dengan mencampurkan Epikote Resin 828 and Hardener 651 dengan nisbah berat campuran 5:2. Keputusan menunjukkan kekuatan tegangan, modulus elastik, tenaga patah Mod I dan Mod II adalah optimum pada 5% TEEP dengan peningkatan masing-masing sebanyak 36.8%, 24.9%, 60.3% and 166.3% berbanding epoksi biasa. Ini disebabkan oleh kekesatan permukaan yang lebih baik dan kelebihan serbuk kulit telur sebagai penahan retakan. Walaubagaimanapun, 10% TEEP menunjukkan penggumpalan serbuk kulit telur yang memberikan ikatan kurang baik dengan epoksi resin dan seterusnya mengurangkan sifat mekanikalnya. Selanjutnya, kekuatan ricih dalam sambungan tindih-tunggal dengan tindihan terpanjang untuk 5% TEEP menunjukkan peningkatan sebanyak 72.7% berbanding epoksi biasa. Kerangka model unsur terhingga telah dibangunkan menggunakan teknik model zon kohesif (CZM) dengan menggunakan hubungan tarikan-pemisahan sebagai model konstitutif. Didapati dengan menggunakan sifat bahan yang ditententuukur memberikan persetujuan yang baik dengan data eksperimen dengan selisih purata kurang daripada 10.8%.

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LIST OF SYMBOLS AND ABBREVIATIONS

a	-	Crack length
A_n	-	Loading angle
B	-	Specimen width
C_o	-	Compliance
ε_x	-	Lateral strain
ε_y	-	Longitudinal strain
E	-	Elastic modulus
E_f	-	Flexural modulus
E_s	-	Tensile modulus
g/m^3	-	Density
G_c	-	Fracture energy
G_{IC}	-	Mode I fracture energy
G_{IIC}	-	Mode II fracture energy
G_T	-	Total energy release rate
h	-	Thickness of adherend
m^2	-	Surface area
P_{max}	-	Maximum load
v	-	Poisson's ratio
σ_o	-	Tensile strength
AIV	-	Aggregates Impact Value
AM	-	Additive Manufacturing
ASTM	-	American Society for Testing and Materials
C0H2D4	-	4-Node Two-Dimensional Cohesive Element
CBT	-	Corrected Beam Theory
CFRP	-	Carbon Fibre Reinforced Plastic
CNSRB	-	Chevron Notched Short Rod Bend
CNT	-	Carbon Nanotubes

CPE4	-	Four-Node Plane Strain Element
CPS8	-	8-Noded Plain Strain Element
CSD	-	Crack Shear Displacement
CSP	-	Coconut Shell Powder
CSR	-	Core Shell Rubber
CTT	-	Compact Tension Test
CZM	-	Cohesive Zone Model
DCB	-	Double Cantilever Beam
DGEBA	-	Bisphenol A diglycidyl ether
DGEBF	-	Bisphenol F diglycidyl ether
DLJ	-	Double-Lap Joint
ENF	-	End-Notched-Flexure
FEA	-	Finite Element Analysis
FEM	-	Finite Element Method
FRP	-	Fibre-Reinforced Plastics
IMP3	-	Third Industrial Master Plan
LEFM	-	Linear Elastic Fracture Mechanics
MCT	-	Modified Compact Tension
MOE	-	Modulus of Elasticity
MOR	-	Modulus of Rupture
PLA	-	Poly Lactic Acid
PP	-	Polypropylene
PPS	-	Polyphenylene Sulphide
PTK	-	Polytriazoleketone
PTS	-	Polytriazolesulfone
SCB	-	Semi-Circular Bending
SCB	-	Single Cantilever Beam
SDEG	-	Scalar Stiffness Degradation
SEM	-	Scanning Electron Microscope
SIMPU	-	Silicon-Modified Polyurethane
SLJ	-	Single-Lap Joint
TEEP	-	Toughened Epoxy with Eggshell Powder
TGAP	-	Triglycidyl P-Aminophenol

TSR	-	Tensile Strength Ratio
UTHM	-	Universiti Tun Hussein Onn Malaysia
UTM	-	Universal Testing Machine
VCCT	-	Virtual Crack Closure Technique
XFEM	-	Extended Finite Element Modelling
XRF	-	X-Ray Fluorescence
11MP	-	Eleventh Malaysian Plan
2-D	-	Two-Dimensional
3-D	-	Three-Dimensional
4ENF	-	Four-Point End-Notched Flexure

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CHAPTER 1

INTRODUCTION

1.1 Research background

Recently, considerable literature has grown up within adhesively bonded joint studies. New experimental methods and numerical models are continually presented in the scientific literature and employed in the construction industry to demonstrate the significance of this joining type. Sugiman *et al.* (2019) have conducted an experimental and numerical method to investigate the effect of media and aging conditions on the durability of adhesively bonded joints, and a significant impact was reported. Besides, an experimental and numerical model of adhesively bonded single-lap joint (SLJ) are based on the failure modes exhibited, load-displacement behaviours, bond-slip relations, and the strain distribution along the bond length at different loading stages was performed by Ungureanu *et al.* (2018). The stress-strain behaviour and strain distributions along the bond length of SLJ is based on 3-Dimensional (3D) Finite Element Modelling (FEM) analysis and showed good agreement with experimental datasets.

The adhesively bonded joints have been widely utilized in numerous industries due to their interesting characteristics, such as excellent resistance under fatigue loading and joining dissimilar materials. Besides, adhesively bonded joints demonstrate lower stress concentrations than mechanically fastened joints such as riveted, welded, and bolted joints (Mariam *et al.,* 2018). According to Ozer (2018), the potential benefits of using adhesively bonded joints are providing more uniform stress distribution along the overlap length due to the absence of holes. A SLJ, as seen in Figure 1.1, tickles the attention of researchers owing to its simple manufacturing method and serves as the basis for structural design and certification of bonded structures.

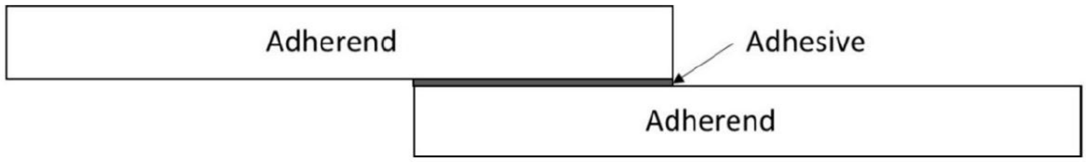

Figure 1.1: The assembly of a single-lap joint (Lempke, 2013)

Epoxy resin is a prominent polymer-type adhesive widely used in construction and civil engineering sectors to join metal-metal materials. Epoxy resin has been certified in other essential applications, such as manufacturing, mechanical and construction sectors. Due to their potential applications in the construction industry, the adhesives strength and joining variables are important and challenging research areas. Nevertheless, most epoxy resins have a severe drawback: their brittleness suggests poor fracture toughness, low impact strength, and low crack propagation resistance (Kuzmina *et al.,* 2022). Its inherent fragility has limited its application in industries requiring high-impact resistance and fracture toughness.

Awareness of environmental concerns has led to reusing bio-degradable materials as fillers in epoxy resin. According to Li *et al.* (2022), adding the finer constituents into a continuous epoxy resin matrix effectively enhances the epoxy resin toughness. The large aerial particle surface in fillers promotes strong bonding reactions within the epoxy resin matrix (Yeasmin *et al.,* 2021). Among the available bio-waste additives, the widely accessible and inexpensive chicken eggshell has good potential as a filler in epoxy resin since it is a daily food waste disposed of in every household. The chicken eggshells have desirable calcium carbonate ($CaCO_3$) content to increase the toughness and associated impact strength. According to Ahmed *et al.* (2021), an eggshell mainly comprises 95% $CaCO_3$ mineral in the form of calcite and organic material of approximately 3.5%.

From fracture mechanics fundamental, there are three failure modes exhibited, Mode I (opening), Mode II (shear), and Mode III (tearing), as shown in Figure 1.2. Mode III, also referred to as tearing mode, is characterized by shear stresses occurring at out-of-plane shear, and prone to less dominant compared to Mode I and Mode II failures. This failure mode is typically entails a tearing or sliding motion parallel to the direction of the applied shear force. In the case of single lap joints, Mode III failure is not seen as the primary loading direction involves tensile or shear stresses along the bond line. In a typical single lap joint, the adherends are attached and primarily subjected to tension or shear stresses within the overlap region. This geometric configuration is not applicable for Mode III presence, which entail shear deformation perpendicular to the joint.

According to Ozer (2018), adhesive joint failure comprised of cohesion within the adhesive layer, interfacial failure between adherends and epoxy resin, and adherend failure. This study focused on Mode I and Mode II failure associated with cohesive failure. A common technique to measure Mode I fracture energy, G_{IC} of adhesive is the Double Cantilever Beam (DCB) test. In this study, DCB specimens are fabricated to measure the fracture energy value of toughened epoxy with eggshell powder (TEEP). In addition, the End-Notched-Flexure (ENF) test is recognized as a standard test to assess and determine fracture energy in shearing Mode, G_{IIC} (also known as delamination toughness subjected to Mode II failure).

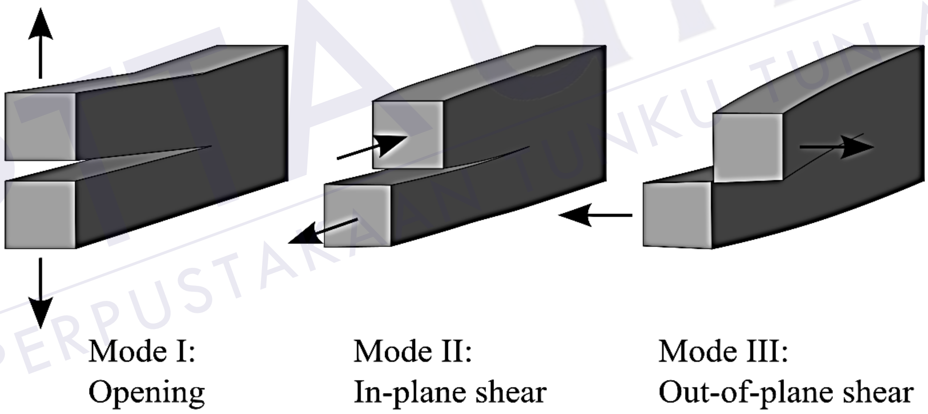

Figure 1.2: Failure modes from fracture mechanics (Oterkus *et al.,* 2016)

To increase confidence in the broader applicability of eggshell powder as filler in epoxy, a reliable numerical model must be developed to perform strength prediction and associated structural response of TEEP in adhesively bonded joints. Hence, numerical analysis was performed using the Finite Element Analysis (FEA) modelling framework. FEA is a powerful tool used to predict the structural response of continuum structures, more favourable in recent years due to the significant evolution in computing technology. Incorporation of physically based traction-separation as a constitutive model can incorporate independent material properties to represent better failure and fracture within cracked bodies investigated. The Cohesive Zone Modelling (CZM) is a numerical technique used to assess the failures of adhesive joints. CZM simulates the fracture process by employing local strength and energy properties to define the delamination process (Carvalho & Campilho, 2018).

The research literature on the incorporation of TEEP in adhesive joints is limited, and an investigation to assess the suitability of eggshell powder as fillers are required. This research aims to measure the material properties and fracture energy of TEEP and evaluate's improvement of TEEP to static shear strength compared to neat epoxy resin. Later, FEA models are developed to perform strength predictions of SLJ toughened with TEEP. The post-processing output from FEA models is validated against the measured experimental dataset, joint variables and associated discrepancies are discussed, respectively.

1.2 Problem statement

According to Moekti (2020), global egg consumption reached 70 million metric tonnes in 2015 and is expected to approach 89 million metric tonnes in 2030, with an annual growth rate of 1.6% from 2015 to 2030. Eggshells are daily food waste disposed of in every household worldwide and the eggshell trash disposed of in landfills has produced environmental issues due to disease growth. Their decomposition has generated an unpleasant odour and microbial growth. Besides, the disposal releases poisonous and damaging gases, including ammonia and hydrogen sulphide, which can harm the environment and lead to human illnesses (Rubright, Peaarce & Peterson, 2017). The eggshells contribute to the sustainable development of building materials as high calcium content in the eggshells can promote high-strength building materials with excellent fracture toughness (Sulaiman *et al.,* 2021).

On the other hand, epoxy resins are commonly utilized in the construction industry. According to Farooq, Teuwen & Dransfeld (2020), epoxy resins have the drawbacks of a longer curing time, low toughness, low impact resistance, and inbuilt brittleness of the cured resin. Farooq *et al.* (2020) concluded that adding fillers to the epoxy resin tends to increase the toughness and strength of epoxy resin. Besides, the modified epoxy resin can increase its shear strength and load-carrying capacity to the applied stress.

The literature mainly reported on using synthetic filler to toughen epoxy resin. The awareness of environmental concerns has led to the reuse of bio-degradable materials as fillers in epoxy resin systems, and the research literature on the incorporation of eggshell powder-filled epoxy is fairly limited. Therefore, a study on TEEP is required to explore these advanced materials' applicability and consider eggshell powder volume fraction and other joint variables, such as the adhesive overlap length. The failure modes and shear strength of adhesively bonded SLJ are evaluated and discussed accordingly.

Due to large arrays involved in adhesively bonded joints, it is difficult to conduct a large volume of experimental testing series resulting in expensive and laborious efforts. The evolution of computing technology promotes accessibility to numerical modelling, especially within the FEA framework. The beauty of FEA modelling is the ability to incorporate a constitutive model (also known as the material model). Formerly, there were difficulties in modelling damage in cracked bodies due to singularity stress ahead of the crack tip. It requires extremely fine meshing, leading to high computational times and difficulty in obtaining convergence. A physically based model, such as the traction-separation relationship driven by the energetic approach, alleviated the difficulties. Traction-separation relationship only requires two material parameters, maximum tensile strength, σ_o and fracture energy, G_c. Previous researchers employed calibrated values of material properties from open literatures. In order to obtain a reliable result, measured material properties from independent testing (or if available in reported literatures, used a subset of materials employed). This allow better strength prediction to enhance the reliability of strength predictions from FEA models developed (Omar, 2023).

1.3 Research objectives

The primary aim of this study is to investigate the tensile properties and fracture energy of TEEP as a function of eggshell volume fractions and later incorporate it within FEA modelling. Static shear strength of adhesively bonded joints is also investigated experimentally and given in the testing series. 2-Dimensional (2-D) FEA models are developed to incorporate explicitly frictional load transfer, surface interactions, and specimen size. Later, the shear strength of adhesively bonded joints

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2. Fong, C., Sugiman, S., Chin, D, D., Ahmad, H. (2023). Fracture Energy and Mechanical Strength of Toughened Epoxy Resin with Eggshell Powder. *Journal of Adhesion Science and Technology*, (submitted– under review).

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