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Static Shear Strength of Single-Lap Joint Using Eggshell-Toughened Epoxy as Adhesive Agent

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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. The shear strength of toughened epoxy with eggshell powder (TEEP) was studied. Single-lap joint (SLJ) shear tests were conducted to investigate the shear strength of the SLJ bonded with different volume fractions of TEEP. For this purpose, the eggshells were dried and crushed into particles of size 150 μ m. The volume fraction of eggshell powder in the epoxy resin is 0%, 2.5%, 5%, and 10% by epoxy weight. The epoxy resin system was made of EPIKOTE Resin 828 and Hardener 651 with a mixing ratio of 5:2 by weight. The results show that the shear strength greatly depended upon overlap length and filler volume fractions. The improvement was up to 72.7% and 39.9%, with the longest overlap length and 5% TEEP fraction. Hence, an overlap length of 38.1 mm and a 5% eggshell volume fraction gave the optimum shear strength of 5.045 kN.

Keywords: Eggshell powder, filler, shear strength, single-lap joint, toughened epoxy

1. Introduction

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. [1] 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 joints based on the failure modes, load-displacement behaviours, stress-strain variation, bond-slip relations and the strain distribution along the bond length at different loading stages were done by Ungureanu et al. [2], the numerical simulations, based on 3D FEM analysis, provided results in good agreement with the experimental.

The adhesively bonded joints have been widely utilized in numerous industries due to their interesting characteristics, such as excellent resistance under fatigue loading and dissimilar joining materials. Besides, adhesively bonded joints demonstrate less stress concentrations than mechanically fastened joints such as riveted, welded, and bolted joints [3]. According to Ozer [4], the potential benefits of using adhesively bonded joints are the provision of different stress distributions along the overlap length and less stress concentrations due to the absence of any cuts or holes. The single-lap joint (SLJ) tickles the attention of researchers owing to its simple manufacturing method and serves as the basis for structural design and certification of bonded structures.

Epoxy resin is a prominent polymer-type adhesive widely used in construction and civil engineering sectors to join metal-to-metal materials. Epoxy has been certified in other essential applications, such as manufacturing, mechanical and construction sectors. Due to their potential applications in the construction industry, adhesive strength and joining variables are important and challenging research areas. Nevertheless, most neat epoxy resin systems have a severe drawback: their brittleness suggests poor fracture toughness, low impact strength, and low crack propagation resistance [5]. Its inherent fragility has limited its application in industries that require high-impact resistance and fracture toughness. According to Li et al. [6], adding the finer constituents into a continuous epoxy resin matrix effectively enhances epoxy adhesive toughness. The large particle surface area of fillers promotes strong bonding with the epoxy resin matrix, improving the mechanical properties of epoxy [7].

The research literature on the incorporation of eggshell powder-filled epoxy in adhesive joints is limited, and an investigation is required to assess the suitability of eggshell powder as filler. The awareness of environmental concerns has led to the reuse of bio-degradable materials as fillers in epoxy resin systems. The widely accessible and inexpensive chicken eggshell has good potential as a filler in epoxy systems since it is a daily food waste disposed of in every household. In Malaysia, 2.8 million eggs are consumed daily [8]. Egg consumption among Malaysian is increasing with high demand for the local food industry. Eggshell waste has a negative impact on the environment and the public's health. Besides, Moekti [9] reported that 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. Sulaiman et al. [10] and Ji et al. [11] stated that eggshell is a promising candidate as a reinforcing agent in the polymer industry and can contribute to the sustainable development of joining building materials. Hence, this research aims to investigate and evaluate the effect of Epikote 828 epoxy toughened with eggshell powder on the static shear strength of SLJ compared to a neat-epoxy system.

2. Experimental Works

This section describes the experimental works conducted to investigate the shear strength of single-lap joints bonded with TEEP. The details for the testing series, selection of adherend, surface preparation of adherend, preparation of TEEP, fabrication of single-lap joint specimen, and single-lap joint shear test are described in the subsequent section.

2.1 Testing Series

The testing series for the single-lap joint shear tests in this investigation are shown in Table 1. A combination of three different overlap lengths, i.e., 12.7 mm, 25.4 mm, and 38.1mm and four-volume fractions of eggshell powder, i.e., 0%, 2.5%, 5%, and 10%, were investigated to give a total of twelve testing series. For each series, three replications experiment works were done to get average results, giving a total of 36 specimens results at final. The series designation follows the sequence of joint type-overlap region-eggshell fractions as given in Table 1.

	e e .		
Volume fraction of eggshell powder (%)	Overlap Length (mm)	Series designations	
	12.7	SLJ-12.7-0	
0	25.4	SLJ-25.4-0	
	38.1	SLJ-38.1-0	
	12.7	SLJ-12.7-2.5	
2.5	25.4	SLJ-25.4-2.5	
	38.1	SLJ-38.1-2.5	
	12.7	SLJ-12.7-5	
5	25.4	SLJ-25.4-5	
	38.1	SLJ-38.1-5	
	12.7	SLJ-12.7-10	
10	25.4	SLJ-25.4-10	
	38.1	SLJ-38.1-10	

Table 1 - Testing series for single-lap shear test

2.2 Selection of Adherend

The single-lap joint test specimen was fabricated according to the ASTM D1002 [12]. Broughton et al. [13] recommended joining identical adherends to minimize skewing of the peak normal stresses and to reduce residual thermal stresses due to differences in the coefficient of thermal expansion values. Low carbon steel (mild steel) was used as adherends. Low carbon steel has low yield strength and is lower in cost.

2.3 Surface Preparation of Adherend

The adherend surface used in adhesive joining plays an important role in allowing stronger and more durable bonding. The excellent quality adhesive joint was performed by roughened steel texture from appropriate surface preparation techniques [14]. The steel surface was roughened using sandpaper and a chemical cleaning method. Amorim et al. [15] state that the roughened surface texture provides better adhesive anchorage to alleviate shear stress. The sandpaper of 120 grit size was used to polish the steel adherends and soaked with acetone solution to eliminate impurity of the adherend surfaces, as shown in Fig. 1(c). To avoid corrosion within the sanding regions, all adhesively bonded joints are fully assembled within 24 hours of the sanding process. The steel adherends are in cool condition during the application of adhesive.



Fig. 1 - Surface preparation of adherend prior to mechanical testing; (a) low carbon steel ready for surface treatment; (b) sanding process using 120 grit size sandpaper; (c) immersed in acetone solution to eliminate impurities; (d) oven-heated under 120°C



Fig. 2 - Degassing of TEEP

2.4 Preparation of TEEP

The epoxy adhesive was prepared by eggshell powder with different volume fractions of 0%, 2.5%, 5%, and 10%. The epoxy resin containing eggshell powder was then stirred for approximately 10 minutes at ambient temperature using a magnetic stirrer to obtain a homogeneous and uniform mixture. After 10 minutes, the hardener was added to the TEEP and stirred for another 5 minutes. The resin to hardener ratio was 5:2 (by weight). The mixing process should be performed slowly to prevent air bubbles from penetrating the mixed epoxy resin system. A faulty mixture process can

cause entrapped voids and poor adhesive performance. Owuamanam et al. [16] suggested using a vacuum chamber for degassing purposes, primarily used in the epoxy resin mixture. The degassing process removed entrapped air bubbles from the epoxy system. The epoxy was degassed for 10 minutes at a vacuum desiccator, as shown in Fig. 2, to remove the entrapped air inside the TEEP.

2.5 Fabrication of Single-Lap Joint Specimen

Fig. 3 illustrates the single-lap joint sample dimensions. The adhesive overlap lengths are 12.7, 25.4, and 38.1 mm. The thickness of adhesive and adherends are 0.2 mm and 2 mm, respectively.



Fig. 3 - Dimensions of single-lap joint configurations according to ASTM D1002

2.6 Single-Lap Joint Shear Test

First, the lines of both steel adherends were drawn to indicate the overlap length. The adhesive was uniformly distributed over the designated overlap region. A catgut string with a 0.2 mm diameter was used to regulate the adhesive bonding thickness. A clip was then used to secure the bonding of the joining adherends. The well-prepared adhesively bonded joints were allowed to cure at room temperature for 24 hours to obtain a strong interfacial contact between the adhesive-adherends layer. Upon 24 hours of curing, the single-lap joint specimens were ready for mechanical testing. Two steel spacers were attached at the end tab prior to testing, as seen in Fig. 4(c), to reduce the bonding moment and ensure that the epoxy bond-line was dominated by shear stress under tensile stresses. The INSTRON universal testing machine was used to analyze single-lap joints. The test specimens are placed in the grips of a universal testing machine and pulled at 0.5 mm/min until rupture, according to ASTM D1002. Upon testing, the images of the failure mode exhibited were recorded for data analysis.



Fig. 4 - Experimental procedures in the single-lap shear test; (a) lines drawn to indicate overlap region; (b) SLJ bonding using TEEP; (c) providing spacers to avoid primary bonding; (d) single-lap shear test set-up

3. Results and Discussions

This section covered the shear strength of single-lap joints toughened with varying eggshell powder volume fractions of TEEP. It started with discussions on load-displacement profiles and associated observations during mechanical testing. Next, the shear strength and failure modes of SLJ were elaborated to concentrate on the effect of overlap length and TEEP volume fractions.

3.1 Load-Displacement Profiles

Fig. 5 depicts the load-displacement curve for a single-lap joint tensile test. It exhibits the characteristics of a single-lap joint under quasi-static loading with a speed load of 0.5 mm/min. Initially, the joints exhibited linear elastic behaviours before fracture and failure initiation at the TEEP adhesive edge propagated towards adhesive layers. Subsequently, the maximum load is exhibited to give a catastrophic failure. An audible screeching sound was heard during mechanical testing to indicate an epoxy fracture after the crack had exceeded a certain length to give maximum load at failure and failed catastrophically. Fig. 5(a) demonstrates an elastic zone followed by a total fracture of the sample, indicating a brittle failure. Fig. 5(b) shows that the tensile strength improves by incorporating eggshell as an additive. The maximum stiffness was found in 5 % eggshell addition, with further increased eggshell concentration prone to tensile stiffness reduction.



Fig. 5 - Load-displacement profile; (a) typical load-displacement of single-lap joint; (b) load-displacement with different incorporation of TEEP volume fractions



Fig. 6 - Sketch of damage plot at key-points labelled in Fig. 5(a)

3.2 Failure Modes and Experimental Observations

From the experimental observations, all single-lap joints with TEEP-bonded demonstrated cohesive failure modes, which denotes visible plate separation with adhesive fragments on both adherends surfaces. By inspection, the failure surfaces of the tested joints revealed a thin adhesive film over the bonded area. A thin layer suggests that the adhesive failure is prone to exhibit cohesive failures with less shear resistance than thicker adhesive counterparts. More considerable shear stress exhibited is the primary cause of cohesion failure, associated with applied shear stress that has exceeded the adhesive's shear strength. As seen in Fig. 7, cohesive failure occurred in the TEEP adhesively bonded single-lap joint because a large crack had completely breached the epoxy bond line, and two split epoxy portions remained on both fracture surfaces. Additionally, the adhesive region has large visible and tiny cracks.



Fig. 7 - Cohesive failure in different adhesive overlap length; (a) 12.7 mm; (b) 25.4 mm; (c) 38.1 mm

3.3 Shear Strength of Adhesively Bonded Single-Lap Joint

Table 2 demonstrates that for every TEEP series, the maximum load increases as the adhesive overlap length increases. Overall, the shear strength ranges between 2.441 - 5.045 kN, where three specimens were prepared for each testing series to allow reproducibility and reliability. The P_{max} increased to 2.725 kN and 2.922 kN with the incorporation of 2.5 % and 5% eggshell volume fraction, respectively, to indicate an improvement of 4.56% and 12.1% as compared to neat epoxy, which has a tensile strength of 2.606 kN.

In addition, the shear strength of single-lap joints is constant up to a volume percentage of 5%; then, it begins to decline. At a high-volume percentage of 10%, adhesives become more viscous and incapable of quickly wetting the adherend surface. The decrease in shear strength with the addition of TEEP at a volume percentage of 10% may result from an excessive filler, which causes agglomeration. As particles are bound together and function as micro-particles, the agglomeration has effects such as a reduction in surface area, which in turn causes stress transmission [17]. Additionally, agglomeration lowers the adhesion between particles and epoxy resin.

Moreover, when the cohesiveness between particles is poor, particle aggregation may behave as a concentrated stress zone, resulting in fast crack propagation. Sugiman et al. [18] found that the fracture surfaces tend to be smoother

with increasing fly ash volume fraction. This indicates that increasing the fly ash content in the epoxy contributes to an increase in the brittleness of the fly ash-filled epoxy to increase elastic modulus. It is suggested that similar behaviours were observed in incorporating TEEP epoxy. However, the microscopic investigation is outside the scope of this project.

Volume Fraction (%)	Overlap length (mm)	Specimen No.	Failure Mode	Shear Strength, P _{max} (kN)	Mean Shear Strength, P _{max} (kN)	Shear strength Improvement with respective to	
						Overlap length	TEEP volume
0	12.7	S 1	Cohesive	2.794	2.606 ± 0.179	Base	Base
		S2	Cohesive	2.587			
		S 3	Cohesive	2.437			
	25.4	S 1	Cohesive	3.306	3.368 ± 0.057	29.2%	Base
		S2	Cohesive	3.379			
		S 3	Cohesive	3.419			
	38.1	S 1	Cohesive	3.432	3.604 ± 0.237	38.3%	Base
		S2	Cohesive	3.505			
		S 3	Cohesive	3.874			
2.5	12.7	S1	Cohesive	2.832	2.725 ± 0.140	Base	4.56%
		S2	Cohesive	2.776			
		S 3	Cohesive	2.566			
	25.4	S 1	Cohesive	4.504	4.285 ± 0.239	57.2%	27.2%
		S2	Cohesive	4.030			
		S 3	Cohesive	4.320			
	38.1	S 1	Cohesive	4.306	4.435 ± 0.144	62.8%	23.1%
		S2	Cohesive	4.408			
		S 3	Cohesive	4.590			
5	12.7	S 1	Cohesive	3.021	2.922 ± 0.191	Base	12.1%
		S2	Cohesive	3.044			
		S 3	Cohesive	2.702			
	25.4	S 1	Cohesive	4.469	$\begin{array}{c} 4.507 \pm \\ 0.150 \end{array}$	54.2%	33.8%
		S2	Cohesive	4.672			
		S3	Cohesive	4.379			
	38.1	S1	Cohesive	5.001	5.045 ± 0.147	72.7%	39.9%
		S2	Cohesive	5.209			
		S3	Cohesive	4.925			
10	12.7	S 1	Cohesive	2.227	2.441 ± 0.194	Base	-6.3%
		S2	Cohesive	2.607			
		S3	Cohesive	2.488			
	25.4	S 1	Cohesive	3.144	3.130 ± 0.064	28.2%	-7.1%
		S2	Cohesive	3.186			
		S3	Cohesive	3.061			
	38.1	S1	Cohesive	3.376	$\begin{array}{c} 3.422 \pm \\ 0.058 \end{array}$	40.2%	-5.0%
		S2	Cohesive	3.487			
		S3	Cohesive	3.403			

Table 2 - Overall shear strength of SLJ bonded with TEEP

The shear strength of single-lap joints bonded with 0% TEEP increases as a function of adhesive overlap length. Compared to the 12.7mm adhesive overlap length, the maximum load in the 25.4mm and 38.1mm adhesive overlap regions increased by 29.2% and 38.3%, respectively. In this study, the overlap length investigated is limited to 38.1mm (relatively shorter). Therefore, the effect from secondary bending is less likely to be pronounced. A similar finding was reported by Raos et al. [19], where the optimum overlap length is 40 mm. Exceeding this length exhibited lower shear strength.

Besides, the increased overlap length promotes more considerable shear strength (given as maximum tensile force, Pmax) due to increased bonding area. This can be seen in Table 2, where shear strength increased with longer overlap lengths. Raos et al. [19] reported that maximum strength was reached at specific overlap lengths of 40 mm. This optimal overlap length leads to maximal overall shear strength. Further increase of the overlap length over this

optimum value has the consequence of load bearing decline of the joint, and adherend exceeds into the plastic region. At this point, equilibrium between stress in adherends and strength of the adhesive joint is achieved. Beyond this point, excessive deformation of the adherends occurs, which cannot be compensated for by the relatively rigid adhesive layer. This leads to failure inside the adherend. Therefore, the maximum tensile force P_{max} decreases.

4. Conclusion

The shear strength greatly depended upon overlap length and filler volume fractions. The optimal shear strength was measured at 5% volume fraction, further increasing prone shear strength reduction resulting from particle agglomerations. Overall, the improvement was up to 72.7% and 39.9%, with the longest overlap length and 5% TEEP fraction. In this study, an overlap length of 38.1 mm and a 5% eggshell volume fraction gave the optimum shear strength of 5.045 kN. All testing specimens showed cohesive failures, partly due to thin adhesive thickness and incorporation of brittle-type adhesive.

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