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Elastic Properties of Toughened Mussel Epoxy Under Aged and Unaged Conditions

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Abstract

This study investigates the mechanical behavior of silane-treated mussel powder modified epoxy (SMPME) composites under dry and hydrothermally aged conditions (60°C, 50 days). The effects of filler volume fraction on tensile properties, including elastic modulus, Poisson's ratio, and tensile strength, were evaluated using dog-bone specimens. Results show that SMPME generally enhances mechanical performance, with a non-linear trend and an optimal filler content. After aging, an overall improvement in properties was observed, attributed to post-curing and improved filler–matrix interfacial bonding. Although absolute values are lower than literature due to fabrication-related factors, the trend remains consistent. Overall, SMPME demonstrates strong potential as a sustainable reinforcement for epoxy composites.

1. Introduction

Epoxy adhesives are widely used in structural engineering due to their high bonding strength and compatibility with concrete, steel, and CFRP systems in infrastructure applications. However, their brittleness and susceptibility to moisture and thermal aging can cause plasticization, stiffness reduction, and interfacial degradation, reducing long-term durability (Rudawska & Frigione, 2020; Ahad et al., 2023).

To improve performance and sustainability, particulate fillers have been introduced to epoxy systems. Mussel shell powder ($CaCO_3$), a bio-waste material, has shown potential as a sustainable reinforcement due to its stiffness-enhancing capability. Silane surface treatment further improves filler–matrix bonding and reduces agglomeration (Li et al., 2022; Gigante et al., 2020).

However, limited studies have investigated silane-treated mussel shell powder modified epoxy (SMPME) under hygrothermal aging. The effects of filler volume fraction on elastic modulus, Poisson's ratio, and tensile strength, as well as property retention after aging, remain unclear. Comparative evaluation with conventional structural adhesives is also insufficient.

This study therefore examines the elastic properties of SMPME under dry and hydrothermally aged conditions across 0–10% filler content. Dog-bone specimens were tested in tension to determine elastic modulus, Poisson's ratio, and ultimate tensile strength. The work is limited to linear elastic behavior and aims to evaluate the mechanical performance and sustainability potential of SMPME for structural applications.

2. Research Methodology

This study investigates the elastic properties of silane-treated mussel shell powder modified epoxy (SMPME) under dry and hygrothermal aging conditions using ASTM D638 tensile testing. The experiment evaluates the effects of filler volume fraction and environmental exposure on stiffness, deformation, and strength, ensuring controlled comparison between variables

Table 1 *Testing series for SMPME volume fraction*

Series Variables	SMPME Volume Fractions (%)	Testing Specimens Fabricated	Conditions	Total Nos. of Specimens
Variation of modified-mussel powder (SMPME) volume fraction	0	Dogbone	Dry, Aged*	6
	2.5	Dogbone	Dry, Aged*	6
	5.0	Dogbone	Dry, Aged*	6
	7.5	Dogbone	Dry, Aged*	6
	10.0	Dogbone	Dry, Aged*	6

2.1 Preparation of Silaned Mussel Epoxy (SME)

The production of silane-treated mussel shell powder (SMP) involves cleaning, drying, crushing, sieving and silane surface modification of raw shells. Each step is essential to obtain fine, uniform particles with improved dispersion and interfacial bonding for enhanced epoxy composite performance under hygrothermal conditions.

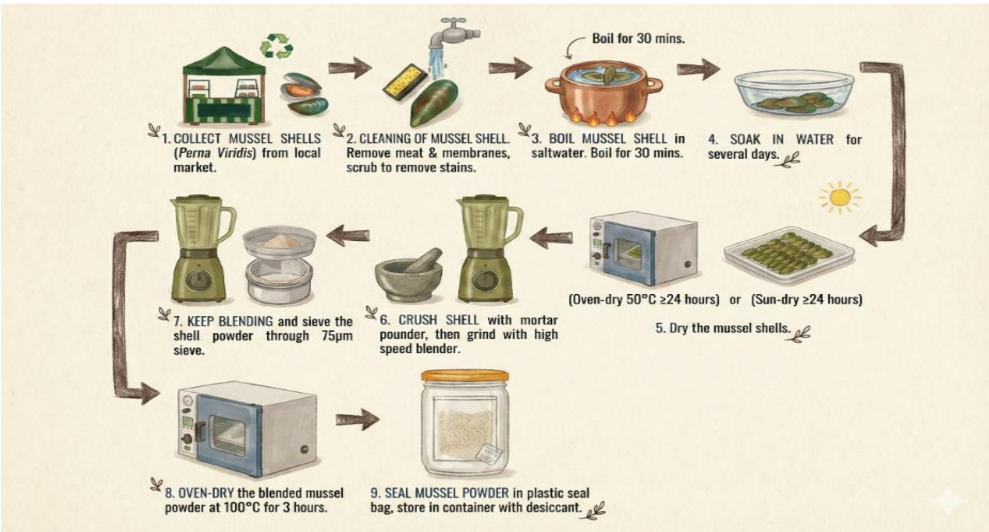

Fig. 1 *Flowchart of mussel powder preparation*

AMPTES (3-Aminopropyltriethoxysilane) was used as the silane coupling agent to modify mussel shell powder. The powder was dispersed in 1-propanol (1:1.3 ratio) and stirred for 20 minutes, followed by addition of AMPTES at a $CaCO_3$-to-silane ratio of 1:0.3. The mixture was then stirred for 2 hours to promote hydrolysis and condensation, forming chemical bonds between the filler surface and epoxy matrix, thereby improving interfacial adhesion and moisture resistance.

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(a) **(b)**

Fig. 2 *Silane solutions (a) 1-propanol; (b) MACKLIN® 3-Aminopropyl triethoxysilane (AMPTES)*

2.2 Fabrication Using Acrylic Molds

Dog-bone specimens were fabricated using ASTM D638-compliant acrylic molds to ensure consistent geometry and smooth surface finish. The epoxy–mussel shell mixture was poured into the molds, gently tapped to remove air bubbles, and left to cure at room temperature. After curing, specimens were demolded and inspected to confirm uniform shape and absence of defects for reliable tensile testing.

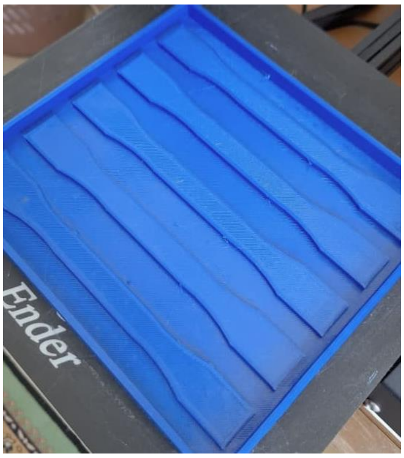


Fig. 3 *Dog-bone specimen mold*

2.3 Preparation for Testing Specimens Under Aged Conditions

Selected specimens were subjected to hygrothermal aging (moisture and elevated temperature) to simulate environmental exposure, allowing water diffusion and associated plasticization and interfacial degradation. After aging, specimens were surface-dried and tested alongside unaged samples to evaluate changes in elastic modulus, Poisson's ratio, and tensile strength.

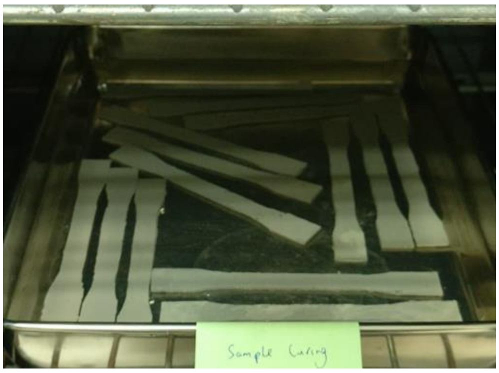
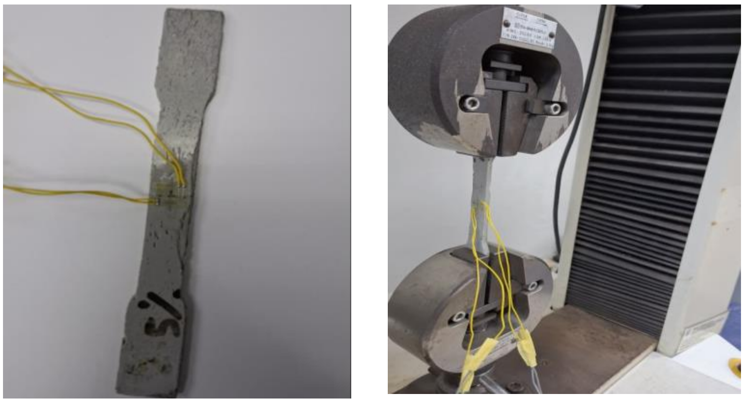


Fig. 4 *Dog-bone specimens under curing*

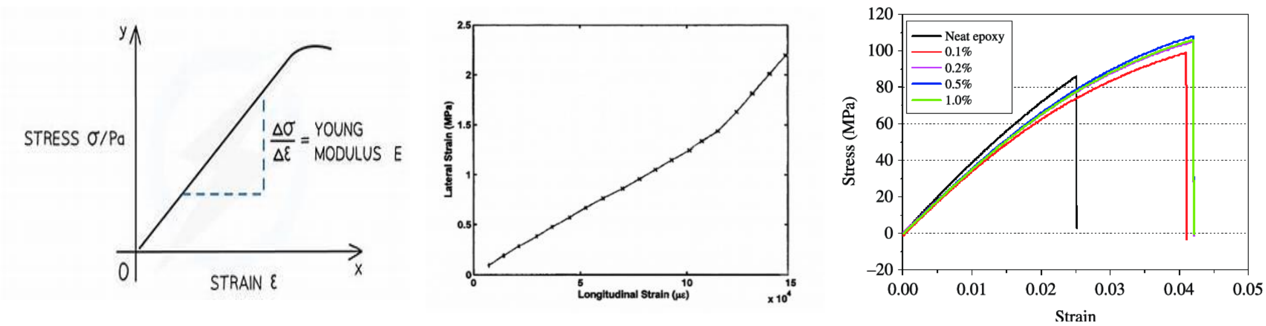
2.4 Experimental Testing

Mechanical testing of SMPME dog-bone specimens was conducted using a Universal Testing Machine (UTM) in accordance with ASTM D638 under both unaged and hygrothermally aged conditions. A uniaxial tensile load was applied at a constant crosshead speed until failure, while a strain gauge bonded at the gauge section was used to obtain accurate axial strain data. End-tab preparation was applied at the gripping regions to prevent slippage and premature grip failure, ensuring fracture occurred within the gauge length. The recorded load–strain responses were used to construct stress–strain curves, from which elastic modulus, Poisson's ratio, and ultimate tensile strength were determined.



(a) **(b)**

Fig. 5 *Testing process (a) Strain gauge; (b) Universal Testing Machine(UTM)*



(a) **(b)** **(c)**

Fig. 6 *(a) Elastic modulus; (b) Poisson's ratio; (c) Tensile stress–strain curves*

3. Results and Discussions

This chapter presents the tensile test results of silane-treated mussel shell powder modified epoxy (SMPME) under both dry and hydrothermally aged conditions. The influence of filler volume fraction on elastic modulus,

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Poisson's ratio, and ultimate tensile strength is evaluated. Comparative analysis is performed to assess environmental effects and structure–property relationships.

3.1 Failure Behaviour

Dry specimens mainly failed within the gauge region, indicating proper load transfer, while occasional crimp-region failure was attributed to gripping effects and stress concentration. Overall, failure behavior reflects a typical brittle polymer response with minor variability due to specimen preparation.

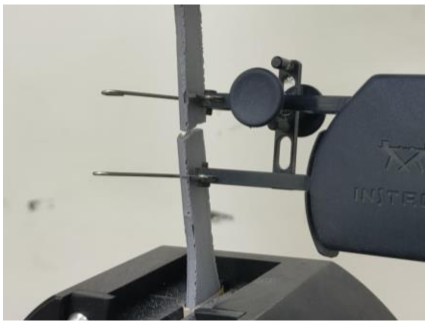

Fig. 9 *Failure at gauge section*

3.2 Elastic Modulus

Elastic modulus increases overall with SMPME content, reaching a peak at intermediate–high filler levels. The improvement is attributed to restricted polymer chain mobility and enhanced stress transfer from matrix to rigid $CaCO_3$ particles.

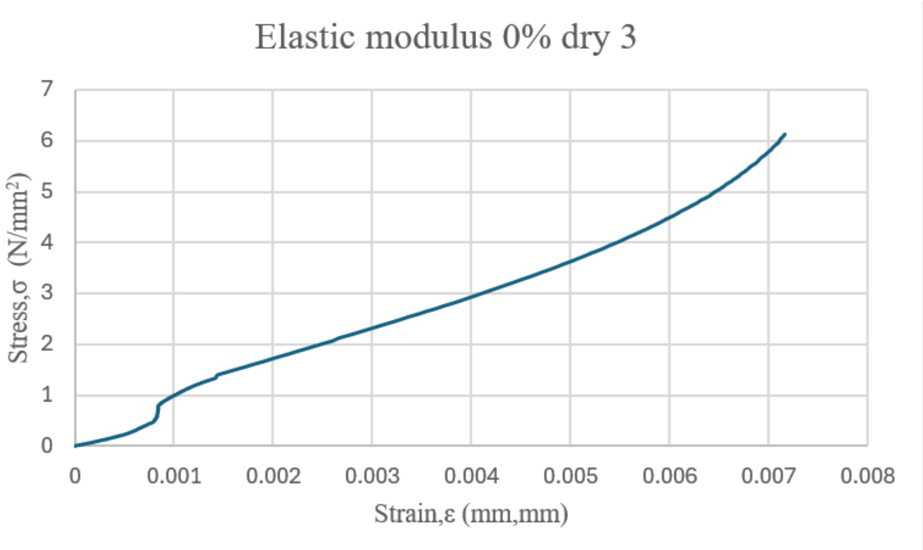

Fig. 10 *Elastic modulus measurement from the stress-strain curve*

Table 2 *Full dataset of measured elastic modulus of aged SMPME*

Volume Fraction (%)	Specimen No.	Elastic Modulus, E (MPa)	Mean Elastic Modulus, E (MPa)
0	S1	920	612.619±290.405
	S2	575	
	S3	342.857	
2.5	S1	1850	1573.529±242.018
	S2	1400	
	S3	1470.588	
5	S1	1428.571	1345.5841±261.530
	S2	1052.632	
	S3	1555.555	
7.5	S1	2608.7	1584.08±887.55
	S2	1052.632	
	S3	1090.909	
10%	S1	1235.294	1755.199±551.331
	S2	2333.333	
	S3	1696.97	

3.3 Poisson's Ratio

Poisson's ratio shows slight fluctuations with filler addition, indicating minor changes in lateral deformation behavior. The overall trend suggests that SMPME does not significantly compromise dimensional stability.

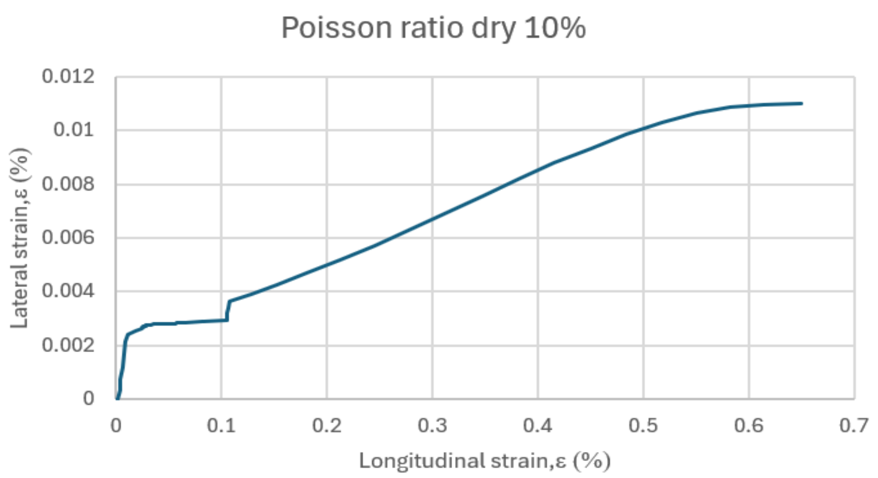

Fig. 11 *Poisson's ratio measurement from lateral strain-longitudinal strain curve*

Table 3 *Full dataset of measured Poisson's ratio of aged SMPME*

Volume Fraction (%)	Specimen No.	Poisson's ratio, v
0	1	0.3513
5	1	0.1661
10	1	0.206

3.4 Ultimate Stress at Failure

Ultimate stress at failure was obtained from the peak of the stress–strain curve to represent the maximum tensile capacity of SMPME composites. Results show a non-linear dependence on filler content due to the balance between reinforcement and particle agglomeration. Aged specimens were compared with dry samples to assess strength retention under hygrothermal conditions.

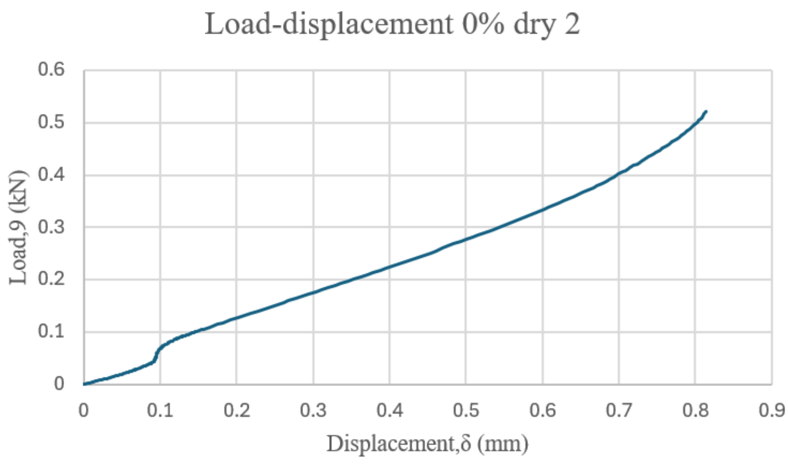

Fig. 12 *Load-displacement profile*

Table 4 *Full dataset of measured tensile strength of aged SMPME*

Volume Fraction (%)	Specimen No.	Maximum Load, P_{max} (N)	Tensile Strength, σ_0 (N/mm^2)	Mean Tensile Strength, σ_0 (N/mm^2)
0	S1	942.7	11.78	8.897±2.506
	S2	613.7	7.67	
	S3	579.3	7.24	
2.5	S1	444.6	5.558	7.389±2.110
	S2	553	6.913	
	S3	775.7	9.696	
5	S1	583.8	6.735	8.605±2.213
	S2	883.8	11.048	
	S3	642.6	8.033	
7.5	S1	982.4	12.28	9.925±2.115
	S2	654.9	8.186	
	S3	744.8	9.31	
10%	S1	1053	13.16	10.147±2.684
	S2	641.1	8.014	
	S3	741.3	9.266	

4. Conclusion

This study investigated the mechanical behaviour of silane-treated mussel powder modified epoxy (SMPME) composites under both dry and hygrothermally aged conditions, with emphasis on the influence of filler volume fraction on stiffness, deformation behaviour, and tensile strength. Under dry conditions, SMPME generally enhances elastic modulus and tensile strength due to improved stress transfer and the rigid nature of the filler, although non-linear trends are observed because of dispersion variability and possible agglomeration effects.

After hygrothermal aging, the composites demonstrate an overall improvement in mechanical performance rather than degradation. This behaviour is attributed to post-curing of the epoxy matrix and strengthened filler–matrix interfacial bonding facilitated by silane treatment. The results confirm that SMPME can maintain structural stability under environmental exposure and exhibits an effective reinforcement window at optimal filler content.

For future work, improvements should focus on fabrication consistency. The use of rigid moulds is recommended to reduce dimensional defects and void formation, while mechanical stirring should replace manual mixing to improve filler dispersion. In addition, adopting a more controlled casting method such as continuous pouring or injection can further reduce air entrapment and improve specimen uniformity, thereby enhancing the reliability and repeatability of results.

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Conflict of Interest

Authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Jason Ling Ji Chung, Hilton @ Mohd Hilton Bin Ahmad;* ***data collection:*** *Jason Ling Ji Chung;* ***analysis and interpretation of results:*** *Jason Ling Ji Chung, Nornashima Binti Md Ali;* ***draft manuscript preparation:*** *Jason Ling Ji Chung. All authors reviewed the results and approved the final version of the manuscript.*

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