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Enhanced Mechanical and Morphological Properties of Surface-Modified Industrial Hemp Preform as Reinforcements in 2D Vinyl Ester Composites for Automotive Interiors

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Abstract **Original Research Article**

This study investigated the influence of surface modification on the mechanical and morphological properties of industrial hemp preforms reinforced in 2D vinyl ester composites produced through the Vacuum-Assisted Resin Transfer Molding (VARTM) technique. Industrial hemp preforms were grafted with methyl methacrylate (MMA) monomer to enhance fibre matrix interfacial bonding. The grafting efficiency (44.2%) and grafting percentage (21.5%) were determined, and the resultant grafted and ungrafted composites were evaluated for tensile, flexural, impact, hardness, and abrasion resistance properties according to ASTM standards. The results revealed significant mechanical enhancement in grafted composites. The tensile and flexural strengths increased from 44.57 MPa and 51.34 MPa (ungrafted) to 69.42 MPa and 85.34 MPa (grafted), respectively, representing approximately 55% and 66% improvements. Impact strength rose from 3.83 kJ/m² to 7.05 kJ/m², while Shore D hardness increased from 80.83 to 90.5, indicating better surface rigidity and interfacial cohesion. SEM micrographs confirmed reduced voids, fibre pull-out, and improved matrix wetting in grafted composites, demonstrating the effectiveness of MMA grafting in improving adhesion and load transfer efficiency. The study concludes that surface modified ihemp preforms exhibit superior performance compared to ungrafted ihemp preform making them promising materials for sustainable, lightweight, and eco-friendly interior components in the automotive.

Keywords: Industrial hemp, Surface modification, Infusion vinyl ester resin composites, VARTM, Automotive interiors.

Introduction

Polymer composites are advanced materials formed by combining polymers with reinforcing agents to achieve superior mechanical, thermal, and chemical properties. These materials are engineered to overcome the limitations of traditional polymers by enhancing stiffness, strength, and dimensional stability while maintaining low weight. The synergy between the polymer matrix and reinforcement phase allows for precise tailoring of properties to meet specific functional requirements. Due to their

design flexibility, corrosion resistance, and cost-effectiveness, polymer composites have become indispensable in diverse engineering sectors, including construction, transportation, energy, and consumer goods. Their adaptability and performance advantages continue to drive innovation toward sustainable and lightweight material solutions.

The global composites market has experienced consistent growth, driven by the increasing demand for lightweight, high-performance, and durable materials across multiple industries. According to Grand View Research (2024), the global composites market size was valued at over USD 120 billion and is expected to expand at a compound annual growth rate (CAGR) of around 6–8% through 2030. This growth is propelled by applications in automotive, aerospace, construction, and renewable energy sectors, where efficiency and weight reduction are critical.

In the automotive industry, composites are used to replace conventional metals in components such as body panels, bumpers, hoods, drive shafts, and interior structures. Their low density and high strength-to-weight ratio contribute to improved fuel efficiency and reduced carbon emissions, (Market Research Future, 2023; Mordor Intelligence, 2024).

Industrial hemp fibres have emerged as a sustainable reinforcement alternative for polymer composites, offering a balance between performance, renewability, and cost-effectiveness. In the automotive sector, hemp-reinforced composites are used in non-structural and semi-structural parts such as door panels, seat backs, dashboards, and trunk liners. Their low density, good mechanical strength, biodegradability, and acoustic insulation make them suitable substitutes for synthetic fibres like glass and carbon (Pickering et al., 2016; Sullins et al., 2017). Car manufacturers such as BMW, Mercedes-Benz, and Audi have incorporated hemp-based composites in interior components to reduce vehicle weight and environmental impact (Joshi et al., 2020).

Materials and Methods

Industrial hemp yarn was purchased from Hemp affairs and woven into plain preform. infusion vinyl ester resin INSPECT 585 M and MEKP were purchased from sigma Aldrich

Equipment used include; Universal Material Testing Machine (Tensile), TM2101-7, Charpy Impact Testing Machine, 412-07-15269c, No:HD 96 QD, Shore D Hardness tester, Mul-pc, 0211, Taber Abrasion Tester, model 1700. Floor loom (AD-a-hardness loom) domiciled at Industrial design ABU Zaria, was used to weave Ihemp yarn into plain preform. Vacuum pump, MBF wooden mould measuring 200 x 150 x 200 cm^2 , catch pot, vacuum bag, peel ply, infusion mesh, line clamp, sealant tape used were purchase from Easy Composite, United Kingdom (UK).

Composite preparation

Grafted and Ungrafted industrial hemp preforms with methyl methacrylate monomer with grafting and efficiency percentages of 21.5 % and 44.2 % respectively were infused with infusion vinyl ester resin using Vacuum Assisted Resin Transfer molding method (VARTM) to manufacture composites using the method described by (Ishikawa *et al.,* 2014).

Both grafted and ungrafted ihemp preforms reinforced composites were produced by stacking separately two plain woven preforms, to obtain a two layered preform of dimension (150 X 100 mm^2) on a flat wooden layer mould of dimension 200 x 150 x 200 cm^2. Care was taken to maintain preforms alignment and structure to avoid wrinkling and lateral movement which can affect the overall performance characteristics of the resultant composites.

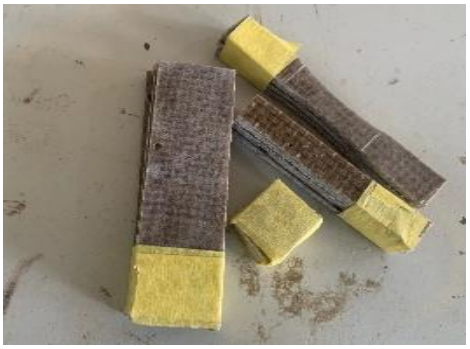
The liquid phase of the matrix forming material which is the uncured vinyl ester resin based on the INSPEC 585 M and Methyl Ethyl Ketone (MEKP) hardener was prepared in accordance with manufacturers recommendation (preform to resin ratio, 60:40, resin to hardener ratio, 2 % of hardener, 50:1) was applied to two layers of each of the grafted and ungrafted Ihemp preforms separately. A 70 x 240 mm^2 infusion mesh was placed on top of the peel ply for resin free distribution evenly across each of the two layer preforms, separately. A 260 x 120 mm^2 peel ply was placed over the assembly to facilitate and ease the removal of the composite from the mould after it is cured. Half an inch-wide sealant

tape with a release paper on side was pressed tightly on the tool around the perimeter of the laminate set-up.

The release paper was peeled off and silicon bag connectors were clamped on both ends of the peel ply facing each other on opposite ends. Nylon vacuum bag was applied on the tape to form a vacuum bag and a port was made to facilitate the application of vacuum pressure to the set-up. Razor blade was used to pierce a small sized square on the nylon vacuum bag directly on top of the silicon connectors to create a space where the vacuum hose for in-let and out let of resin, sealant tape was wrapped around the hose to prevent air out let and maintain vacuum. The vacuum bag completely sealed and to confirm vacuum in the system, a test run of the set up was carried out. Vacuum was drawn and the bag collapsed at a pressure of (-30) psi, indicating a vacuum and air tight system without leakage.

Vinyl ester resin was run on each of the samples of grafted ihemp preform with MMA, ungrafted industrial hemp preforms. Excess resins was deposited into the catch pot housing a small cup design for collecting excess resin directly from the vacuum bag during infusion process. After the vinyl infusion resin gelled, the composites were allowed to stand for 24 hours for complete curing at room temperature.

Figure 1a. VARTM process



1b. Manufactured Ihemp composites

Tensile Strength Test:

Three samples each of grafted and ungrafted industrial hemp composites in dumb-bell shapes as required in accordance with ASTM D638 method for tensile testing of polymer based composite materials were clamped individually at separate intervals between the upper and lower jaws of the universal tensile testing machine which was loaded with 5 KN cells with cross head speed of 4 mm/minute.

The samples were tested three times each and the mean average value was calculated and recorded. The tensile strength at break, tensile modulus and elongation at break were calculated from the following equations:

Tensile Strength = $Breaking \frac{force}{Original} cross\ sectional\ area.$

Tensile modulus = $\frac{Stress}{Strain}$

Elongation at break = $\frac{\Delta l}{\mathrm{L}}\ x\ 100$

where ΔL is the change in the original length and L is the original gauge length

Flexural Strength Test:

Modulus of rupture popularly known as flexural strength, bend strength or even fracture strength is a popular mechanical test for polymer composites based materials. A three-point bending test was carried out according to ASTM D790. The test was conducted using universal material testing machine loaded with 10 KN cell. Three (3) rectangular beam samples each of grafted and ungrafted kenaf and industrial hemp composites samples at a support span length of 44.8 mm and beam length of 59 mm. beam thickness was 2.9 mm and beam width was 12.65 mm. the samples were loaded in 3-point bending as simply supported beam.

The flexural strength and the flexural modulus were determined using the following equations.

Flexural Strength $=\frac{PL}{2bt^2}$

Flexural Modulus $= \frac{PL^3}{4bt^3w}$

Where, L is the span length of the sample. P is the load applied, b and t are the breath and thickness of the specimen respectively and w is the deflection.

Impact Strength Test:

The Impact test of Ihemp grafted and Ungrafted composites were carried out using the Charpy impact testing machine (Norwood instrument Cat-Nr-412-07-15269C) and equipped with 4 joules capacity. The tests were conducted according to ASTM D256.

Hardness Test:

Type D durometer hardness tester was utilized in measuring hardness using the shore D scale according to ASTM 2240. The machine consists of an indenter with a graduated circular tube and a flat surface were samples to be tested are laid. The samples of grafted and ungrafted ihemp composites were place separately at different intervals, the flat surface and the indenter was forced on the surface of the specimen, the load was maintained at a maximum time of 10 to 12 seconds. The test was repeated three (3) times for each of the sample.

Abrasion Resistance Test:

An abrasion resistance test was carried out using a Taber abrasion tester (Rotary Platform Abraser, Model 1700, USA). The test followed ASTM D3389, which evaluates the abrasion resistance of polymer composites by measuring either weight loss after a fixed number of abrasion cycles or the number of cycles to a defined failure point, such as exposure of the underlying sample. Samples of grafted and ungrafted and ihemp composites were cut into 3 mm sections. CS-17 abrasive wheels were installed on the Taber abraser, and the load on each wheel was initially set at 500 g, then adjusted to 1000 g for the composite samples. The vacuum suction was connected to remove debris from the Taber abraser during testing. The abrasion rotation speed was set at 60 rpm as the CS-17 wheels abraded the surface in opposite directions, simulating multidirectional wear. The machine was stopped after 1000 cycles, and the samples were removed. A soft brush was used to clean off debris, and the samples were re-weighed to the nearest 0.1 mg, with the final mass recorded. The test was repeated three times for each of the grafted and ungrafted ihemp composites samples,

Scanning Electron Microscopy (SEM)

To examine the surface morphology of the ungrafted and Ihemp preforms, and to access the uniformity of grafting of MMA monomer and degree of preform coverage, SEM is used. Thermo Fisher Prisma E9954043 SEM testing analyzer machine was used. Samples of grafted and ungrafted Ihemp composites samples were wrapped in gold tape in several portions and images were captured at different magnifications.

Results and Discussion

In this work, the control is the ungrafted ihemp composites (UIH, Ungrafted Industrial hemp, GIH-MMA, Grafted industrial hemp with methyl methacrylate monomer)

Tensile Strength

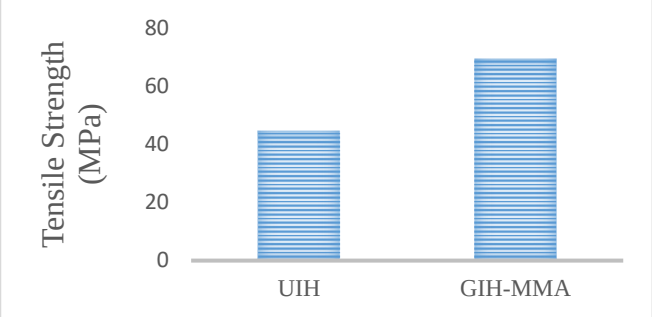


Figure 2: Tensile strength of Ungrafted and grafted Ihemp composites

Figure 2 illustrates the tensile strength of both grafted ihemp with MMA monomer and ungrafted ihemp composites. Ungrafted industrial hemp composite exhibited a tensile strength of 44.57 Mpa. The lower tensile strength result can be attributed to slight reduction of the preform fibre packing density and stress transfer within the vinyl ester matrix. This is supported by similar findings from Pickering *et al.,* (2016), Farouk *et al.,* (2012) where he tensile strength value aligns with known values of ungrafted natural fibre composites which is in the range of 30-60 MPa.

MMA grafted Ihemp composite showed significant improvement in tensile strength with a value of 69.42 Mpa, This is as a result of improved grafting percentage of 44.2 % and pretreatment with sodium hydroxide which created a reactive site on the ihemp preform surface thus enhancing generation of free radicals and subsequently leading to successful grafting. This led to improved interfacial bonding facilitated by efficient stress transfer that is evident in the increased tensile strength. This is supported by findings reported by Kaith *et al.,* (2009) and Mohanty *et al.,* (2000) that grafting can enhance tensile strength by 10-30 %. Study by Farouk *et al.,* (2012), suggest that VARTM process further optimizes fibre wetting and ensure uniform matrix distribution.

Flexural strength

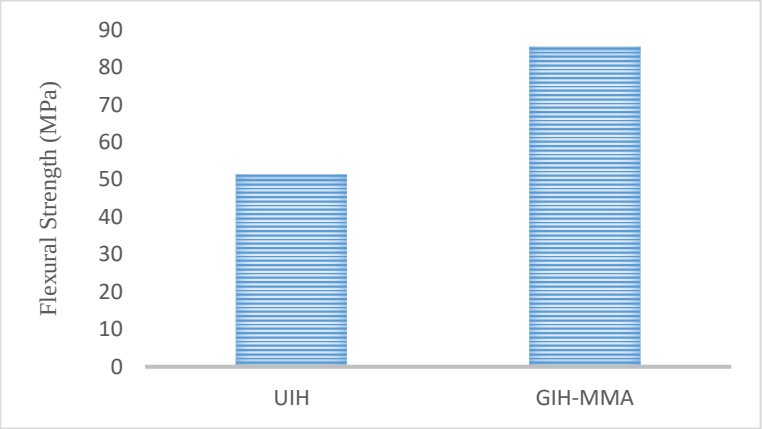


Figure 3: flexural strength of Ungrafted and grafted Ihemp composites

Ungrafted Industrial hemp preform contained impurities such as pectin, lignin, and waxes which naturally inhibit strong interfacial adhesion between preform fibres and polymer matrix. Ihemp ungrafted composite achieved a moderate flexural strength of 51.34 MPa. This result aligns with similar study by Pickering *et al.*, (2016) reported that untreated hemp fibre reinforced composites typically show flexural strength within the range of 40-55 MPa depending on processing conditions and matrix type. The VARTM process used in this work showed effectiveness in reduced low void content. However, Industrial hemp composite grafted with MMA achieved a flexural strength of 85.34 MPa. Grafting parameters of GE 21.5%, and GP 44.2 %, indicated effective grafting process in which a significant portion of the MMA monomer formed covalent bond linkages with preform surface. John and Thomas (2008) in their study of biofibres and biocomposites explained that improved fibre-matrix adhesion minimized voids and reduced fibre pull-out during flexural deformation, resulting in enhanced load transfer. MMA introduced polar ester functional groups on the preform surface which enhanced compatibility with infusion vinyl ester matrix which led to increased interfacial surface energy. These values indicated a highly effective grafting process in which a significant portion of the monomer formed covalent linkages with the preform fibre surface.

Impact strength

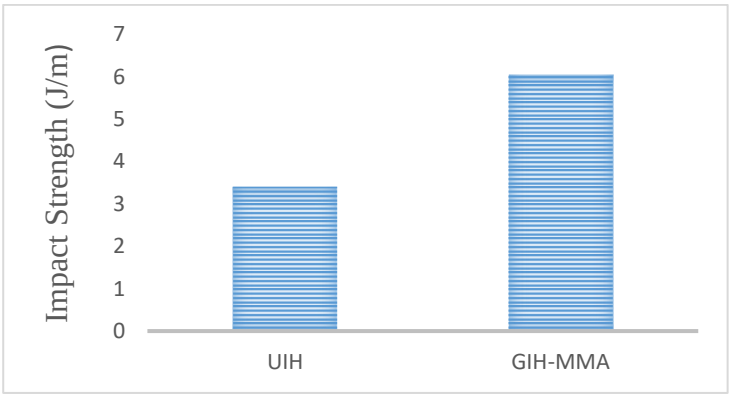


Figure 3: Impact strength of Ungrafted and grafted Ihemp composites

The impact strength is a measure of the energy needed to break the sample on impact. Ungrafted industrial hemp composite gave an impact strength of 3.83 kJ/m². but still reflectes weak interfacial adhesion typical of untreated natural fibres as reported in a similar work by (Kabir et al., 2012). Grafting markedly improved performance, with impact strengths of 7.05 kJ/m² (MMA). MMA grafting achieved higher grafting efficiency (44.2 %) and hence, superior bonding resulting in better stress transfer and energy absorption under impact loading (Zhang et al., 2018).

Hardness

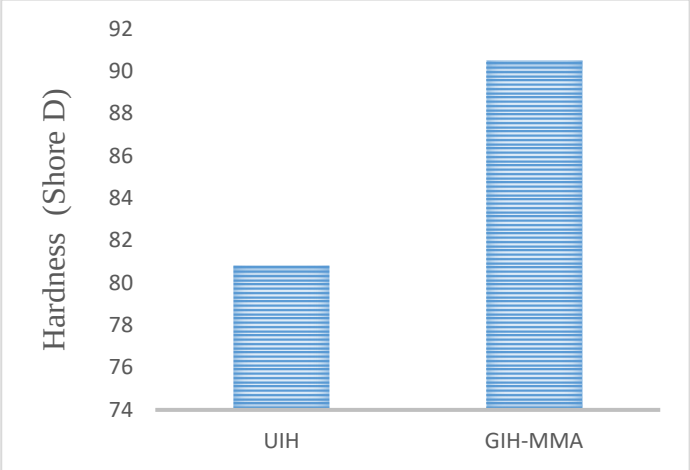


Figure 4: Shore D hardness of Ungrafted and grafted Ihemp composites

Hardness is the resistance of a material to deformation, indentations or scratching. The tests were performed to determine the hardness of all samples using the Shore hardness tester. Ungrafted ihemp composite gave a Shore D value of 80.83, which falls within the typical range for untreated or ungrafted natural fibre composites. Research findings by Sha *et al.* (2016) reported Shore D hardness values for untreated hemp fibre composites in the range of 75–85. Additionally, Bledzki and Gassan (1999) explained that fibre alignment, compaction, and the absence of micro-voids in a matrix processed via vacuum-assisted methods significantly contribute to high hardness values. The high hardness observed for the ungrafted ihemp composite, despite the absence of grafting and pretreatment, can thus be attributed to the VARTM technique used in manufacturing. However, grafted ihemp composites with MMA gave improved Shore D hardness value of 90.5. This improvement is attributed to the successful grafting of MMA monomer onto the surface of the ihemp preform. This modification improved surface roughness and interfacial adhesion. The covalent bonding of MMA chains onto the ihemp preform fibre surface altered the fibre's surface energy and improved compatibility with the hydrophobic vinyl ester resin used in VARTM process. The enhanced interfacial bonding reduced micro-cracking and fibre pull-out under load, thereby increasing surface hardness.

Abrasion Resistance

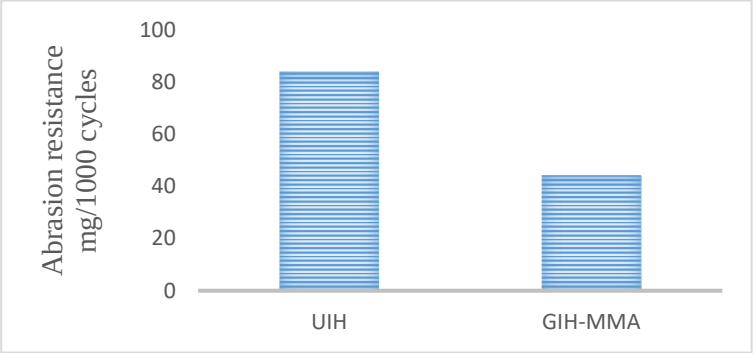


Figure 5: Abrasion resistance of Ungrafted and grafted Ihemp composites

Ungrafted industrial hemp composite recorded a weight loss of 84.0 mg, indicating poor abrasion resistance. This could be attributed to the lack of surface modification of the industrial hemp preform prior to composite manufacturing, which led to poor surface adhesion between the preform fibres and the matrix. This weak interface resulted in fibre pull-out and subsequent matrix wear. A review by Islam et al. (2024) reported that untreated natural fibres often lead to composites with poor wear resistance. Webo (2018), in a study on sisal fibre epoxy-reinforced composites, found that untreated fibres exhibited poor fibre matrix adhesion, causing fibre pull-out. However, MMA-grafted industrial hemp composite recorded a much lower weight loss of about 44.3 mg, representing about 47 % reduction compared to the ungrafted industrial hemp composite. This result indicates superior abrasion resistance. The grafting efficiency of about 44.2 % provided a uniform hydrophobic coating on the ihemp preform surface, which enhanced adhesion and increased surface hardness, thereby reducing wear. This improvement is supported by Islam et al. (2024), where he highlighted that chemical treatments such as acylation, acrylonitrile grafting, and stearic acid enhance the properties of natural fibre composites.

SEM

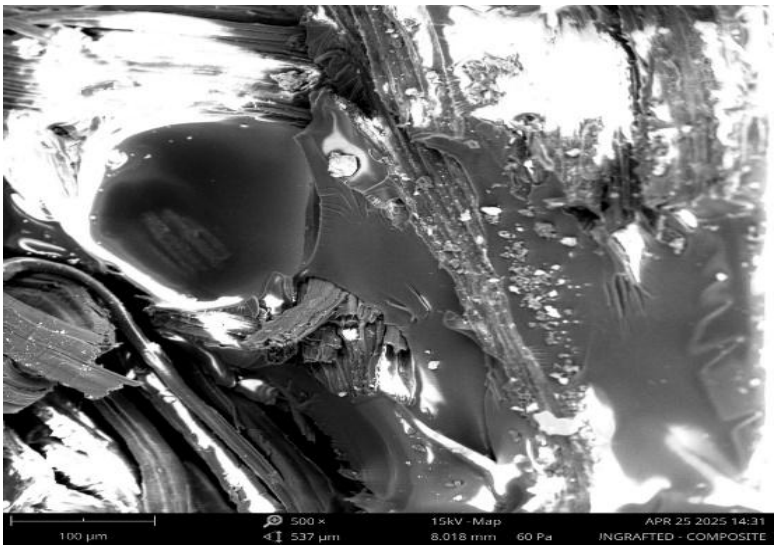


Plate 1: SEM micro-graph of Ungrafted Ihemp composites (x 200)

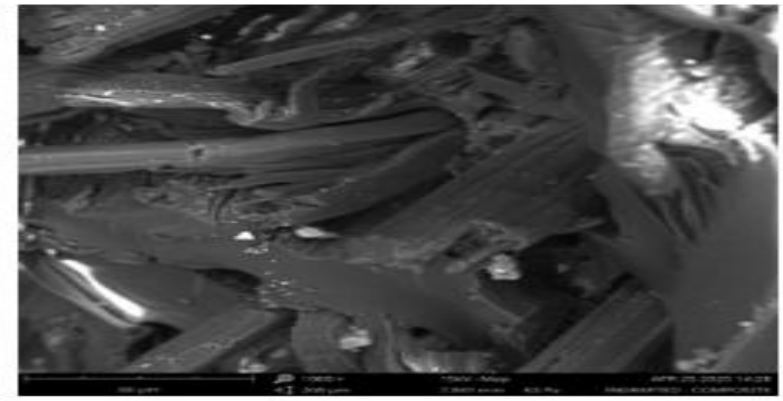


Plate 2: SEM micro-graph of grafted Ihemp composites (x 200)

Plate 1: Depicts the SEM micrograph of the ungrafted industrial hemp composite, revealing fibrous structures with hemp preforms embedded in the vinyl ester matrix. The preforms appear as elongated, cylindrical fibres with rough surfaces and natural impurities such as lignin and hemicellulose. The fibre matrix interface exhibited gaps and fibre pull-out, indicating poor adhesion between the hydrophilic hemp fibres and the hydrophobic vinyl ester resin. Small voids and irregularities further suggest incomplete resin wetting. Similar SEM observations were reported by Thakur et al. (2014).

Plate 2: Depicts SEM micrograph of MMA grafted industrial hemp composites, showing enhanced fibre matrix adhesion compared to the ungrafted sample. The MMA-grafted preform, with a grafting efficiency of 44.2 %, exhibited a thin but effective polymer layer that improved compatibility with the vinyl ester matrix, this is consistent with findings by Liu *et al.* (2018). This also aligns with reports by Thakur et al. (2014) on acrylic grafting improving fibre matrix interfacial properties. Such modifications reduced interfacial gaps and voids and improved resin wetting, likely due to the hydrophobic nature of the grafted layers, thereby enhancing the mechanical properties of the composites.

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