

Microwave-assisted CBS functionalisation of aramid fabric: Surface characterisation and peel adhesion performance in natural rubber composites

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ABSTRACT

Low interfacial adhesion between polar aramid fibres and non-polar elastomeric matrices remains a major challenge in the development of high-performance rubber–fabric composites. In this study, microwave-assisted CBS functionalisation was employed to enhance the interfacial adhesion between woven Kevlar® fabric and natural rubber (NR) composites. The aramid fabric was initially rubberised using an EPDM masterbatch containing different concentrations of N-cyclohexyl-2-benzothiazolesulfenamide (CBS) and subsequently subjected to controlled microwave irradiation prior to composite fabrication. The adhesion performance of the microwave-functionalised composites was evaluated using the ASTM D413 90° strap peel test, while Fourier Transform Infrared Spectroscopy (FTIR), X-ray Photoelectron Spectroscopy (XPS), Thermogravimetric Analysis (TGA), and Scanning Electron Microscopy (SEM) were employed to investigate the chemical, surface, thermal, and morphological characteristics of the treated aramid fabric. The peel response increased from the untreated control to the treated conditions, remained nearly unchanged between E1 and E2, increased further through E3 and E4, and then decreased at E5. The highest mean maximum peel load was observed for E4, corresponding to an improvement of approximately 38% relative to the untreated control. FTIR and XPS analyses provided complementary evidence of changes in the treated/rubberised fabric surface chemical environment and the presence of sulphur-containing treatment-derived species associated with the retained interfacial treatment layer, whereas TGA revealed that the treatment preserved the inherent thermal stability of the aramid fibres. SEM observations further demonstrated increased surface irregularity of the treated/rubberised fabric and improved rubber retention, indicating enhanced fibre–rubber interfacial interaction. Overall, the present results demonstrate the potential of microwave-assisted CBS treatment for improving the interfacial adhesion of aramid fabric–reinforced natural rubber composites under the investigated laboratory conditions.

1. Introduction

Fabric-reinforced rubber composites are widely used in engineering applications where the textile reinforcement provides strength and dimensional stability, and the elastomeric matrix provides flexibility, resilience and energy absorption. Aramid reinforcements are of

particular interest for demanding rubber products such as tyres, hoses, belts, cables and conveyor systems, where mechanical performance, fatigue resistance and reliable fibre-rubber adhesion are of importance [1,2]. The interfacial adhesion between the elastomeric matrix and the reinforcing fabric is of great importance for the overall performance and durability of these composites. Aramid fibres, especially Kevlar, are

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desirable as reinforcement materials, due to their high tensile strength and modulus, low density, good impact resistance and favourable thermal stability [3,4]. These properties are due to the highly orientated aromatic polyamide chains and the strong intermolecular interactions typical of para-aramid fibres. However, the relatively inert and smooth surface of untreated aramid fibres limits effective interaction with elastomeric matrices. Poor aramid-rubber adhesion in natural rubber systems may limit the transfer of stress across the interface and hence reduce the effective utilisation of the reinforcement [5,6].

To overcome this limitation, many surface modification techniques have been investigated to improve the adhesion characteristics of aramid fibres. The conventional chemical treatments are intended to impart reactive functional groups onto the fibre surface or to increase the surface roughness, thereby improving the mechanical interlocking and chemical bonding with the rubber matrix. Using several methods such as plasma treatment, acid etching, alkaline hydrolysis, ultrasonic treatment, polymer coatings, and oxidation processes, improvements were observed in the fibre wettability and the interfacial adhesion. Plasma-based surface treatments have also been widely investigated as an effective route for modifying fibre-surface chemistry and morphology and thereby improving fibre-matrix interfacial adhesion. Recent work by Şenol et al. demonstrated that atmospheric plasma activation of carbon-fibre fabrics increased surface roughness and introduced oxygen-containing functional groups, resulting in enhanced fibre-matrix adhesion and improved tensile and interlaminar fracture performance of carbon-fibre/epoxy composites. Such observations provide further evidence that controlled plasma-assisted surface activation can improve interfacial performance through combined physicochemical modification of reinforcement surfaces [7].

Surface functional groups such as hydroxyl, amino and carboxyl groups have been successfully introduced to increase fibre-matrix interactions. However, many of these treatments involve aggressive chemicals or complex processing conditions that can damage the fibre surface, reduce tensile properties, increase processing cost and limit industrial implementation. Therefore, the development of a simple and effective surface modification technique to improve adhesion without affecting the intrinsic properties of aramid fibres is still an important challenge [8]. Recently it has been shown that controlled surface functionalisation can greatly improve the interfacial performance of fibre reinforced composites without severe structural degradation. In particular, microwave-assisted processing has been identified as an attractive alternative as it provides rapid, volumetric and energy efficient heating while minimising excessive thermal damage to the substrate. Microwave irradiation can enhance chemical interactions at the fibre surface and promote the formation of functional interfaces under relatively mild processing conditions. Moreover, the efficiency of the surface modification process is strongly dependent on the selection of an appropriate functionalisation agent [9]. Vulcanisation accelerators are an interesting possibility among the available functionalisation agents because they are intrinsically compatible with the rubber compounding chemistry. N-cyclohexyl-2-benzothiazolesulfenamide (CBS) is a widely used delayed sulfenamide accelerator which is potentially capable of participating in interfacial reactions and of remaining compatible with sulphur vulcanised rubber systems. It is suggested that CBS could be involved in the formation of an active interface between aramid fibres and the rubber matrix in the presence of microwave irradiation so improving adhesion without harsh chemical treatments. However, there is a lack of studies on microwave-assisted CBS functionalisation of aramid fabrics for enhanced fibre-rubber adhesion, particularly in the case of natural rubber composites [10].

In this study, woven Kevlar fabric was functionalised by microwave irradiation in the presence of CBS incorporated via an EPDM master batch carrier. Then functionalised fabrics were embedded in natural rubber composites and the influence of CBS concentration on interfacial adhesion of fibres with rubber was systematically evaluated by the ASTM D413 strap peel adhesion test. The aim of the study is to develop a

simple, and efficient surface modification strategy to improve adhesion between aramid fabric and natural rubber, while avoiding the disadvantages of conventional chemical surface treatments. The results are expected to be useful for the development of durable high-performance rubber-fabric composites for demanding engineering applications.

2. Materials and methods

Fig. 1 is a schematic representation of the experimental procedure used in the present study. Woven aramid fabric was prepared for the study. Microwave assisted CBS functionalisation was carried out using EPDM as a carrier. Aramid fabric reinforced natural rubber composites were fabricated by compression moulding. Interfacial adhesion between the fibre and rubber was determined using ASTM D413-90° strap peel test. The following subsections discuss the materials, the experimental procedures, the composite fabrication process and the testing methodology [11].

2.1. Materials

Commercial woven Kevlar® 49 aramid fabric (Style 284, 2/2 twill weave) having a nominal thickness of 0.37 mm and an areal density of 170 g m^{-2} was employed as the reinforcing material throughout this investigation. Natural rubber (NR) was used as the matrix material, whereas ethylene propylene diene monomer (EPDM) rubber was employed as an elastomeric carrier phase for CBS, sodium carbonate, and 6PPD during rubberisation and subsequent microwave-assisted treatment of the aramid fabric. No separate removal step was performed after microwave treatment; therefore, the treated rubberised fabric, including the EPDM-based interfacial carrier phase, was subsequently incorporated between the NR sheets during composite fabrication. EPDM was not added as a bulk compounding ingredient to the NR matrix. Accordingly, the resulting adhesion response represents the combined effect of the microwave-assisted CBS/EPDM interfacial treatment under the investigated conditions. Microwave-assisted surface functionalisation was carried out using N-cyclohexyl-2-benzothiazole-sulfenamide (CBS) as the functionalisation agent, sodium carbonate as the catalyst, and N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD) as the antioxidant. The natural rubber compound was prepared using a conventional sulphur vulcanisation system comprising zinc oxide, stearic acid, N660 GPF carbon black, TBBS, DPG, sulphur, and PVI. The Kevlar® fabric was procured from Marktech Composites Pvt. Ltd., Bengaluru, India, whereas CBS, sodium carbonate, and 6PPD were purchased from Sigma-Aldrich, Chennai, India. All chemicals were of commercial grade and were used as received without further purification. The materials employed in the present investigation are summarised in Table 1 [12,13].

2.2. Preparation of microwave-functionalised aramid fabric

The woven Kevlar® 49 aramid fabric was initially cleaned to remove surface contaminants that could interfere with the functionalisation process and subsequent fibre-rubber interfacial interaction. The fabric sheets were thoroughly washed with acetone, rinsed with deionised water, and dried in a hot-air oven at 70°C for 2 h to eliminate residual solvent, moisture, and loosely adhered impurities. Following the cleaning process, the dried fabrics were rubberised using an EPDM-based masterbatch, which served as the carrier phase for distributing the CBS-containing treatment system over the aramid fabric. The EPDM masterbatch consisted of EPDM as the base elastomer together with N-cyclohexyl-2-benzothiazolesulfenamide (CBS) as the functionalisation agent, sodium carbonate as the catalyst, and N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD) as the antioxidant. To investigate the influence of the functionalisation level on fibre-rubber adhesion, the CBS concentration was systematically varied while maintaining all other constituents constant. The compositions of the EPDM masterbatches

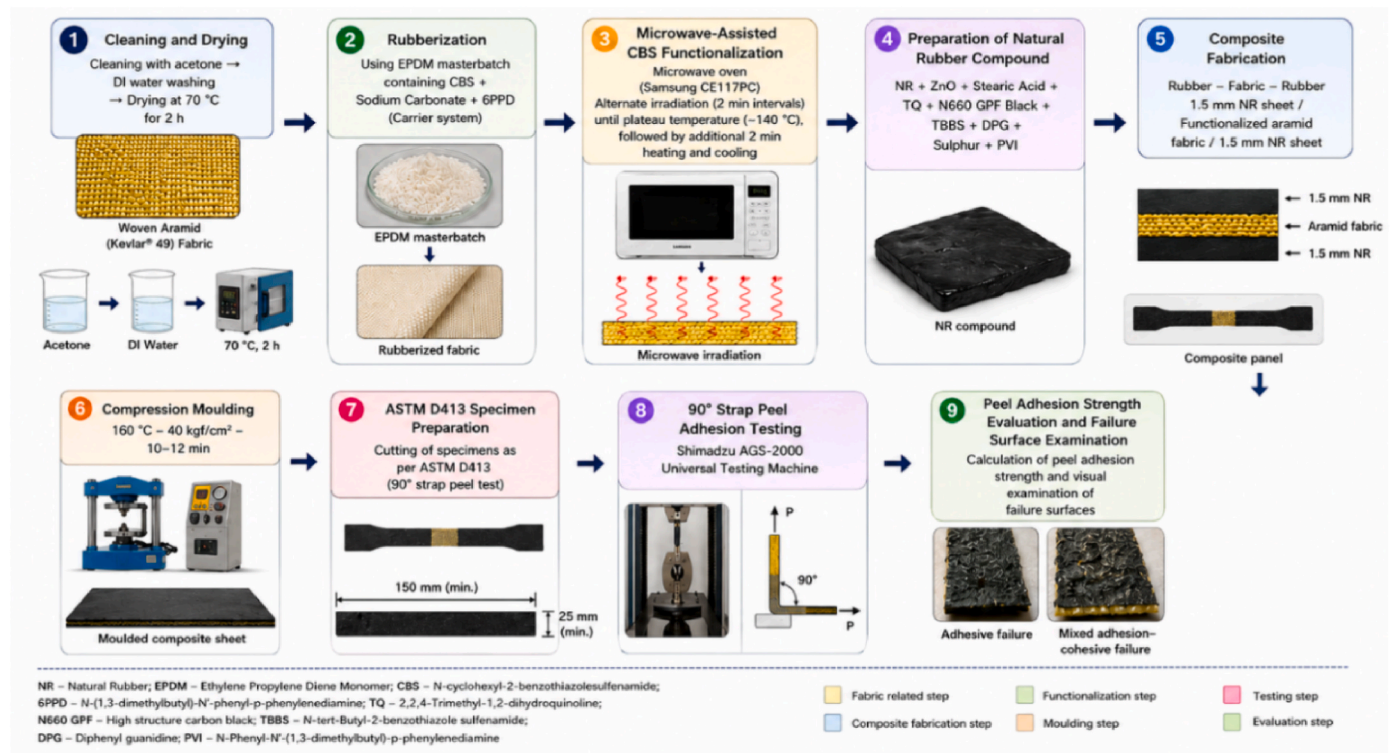


Fig. 1. Experimental workflow illustrating microwave-assisted CBS functionalisation of aramid fabric, composite fabrication, and fibre-rubber interfacial adhesion evaluation.

Table 1

Materials employed in the fabrication of microwave-functionalised aramid fabric reinforced natural rubber composites.

Material	Grade/Type	Function
Kevlar 49® fabric	2/2 Twill weave	Reinforcement
Natural rubber (NR)	Commercial	Matrix
EPDM	Commercial	Carrier rubber
CBS	Sulfenamide	Functionalisation agent
Sodium carbonate	Analytical	Catalyst
6PPD	Commercial	Antioxidant
ZnO & Stearic acid	Commercial	Cure activators
N660 Carbon black	GPF	Reinforcing filler
TBBS, DPG, Sulphur & PVI	Commercial	Vulcanisation system

employed in the present investigation are presented in Table 2.

For consistency, CBS loading throughout the present study is defined as the mass of CBS incorporated per 100 g of EPDM in the functionalisation masterbatch. Accordingly, the E1, E2, E3, E4 and E5 treatment conditions contained 5.6, 11.2, 16.8, 22.4 and 28.0 g CBS per 100 g EPDM, respectively. The EPDM, 6PPD and sodium carbonate contents were maintained constant, while only the CBS loading was systematically varied among E1–E5. Also, the treated fabrics after rubberisation had dimensions of 300 × 150 mm with an average thickness of 0.8 mm and were then subjected to microwave assisted functionalisation. Furthermore, the increase in thickness from approximately 0.3–0.37 mm

Table 2

Formulation of EPDM masterbatches used for aramid fabric functionalisation, showing the CBS mass employed for each treatment condition (E1–E5).

Ingredient (g)	E1	E2	E3	E4	E5
EPDM	100	100	100	100	100
6PPD	15	15	15	15	15
Sodium carbonate	20	20	20	20	20
CBS loading (g per 100 g EPDM)	5.6	11.2	16.8	22.4	28.0

for the original aramid fabric to approximately 0.8 mm after rubberisation resulted from the EPDM-based carrier layer retained on and within the fabric structure [14]. The EPDM-based masterbatch functioned as the carrier phase for distributing the CBS-based functionalisation system over the aramid fabric. Following rubberisation, the fabric was subjected directly to microwave treatment without a separate EPDM-removal step. Thus, the resulting rubberised and microwave-treated fabric was used directly for subsequent NR composite fabrication. Moreover, the untreated aramid fabric without EPDM/CBS treatment and microwave irradiation was employed as the control specimen. The E1–E5 specimens represent the complete microwave-assisted CBS/EPDM treatment system with progressively varied CBS loading. It should therefore be noted that the present experimental design was intended to evaluate the overall effectiveness of the combined treatment and the influence of CBS loading within this treatment system, rather than to independently isolate the individual contributions of EPDM, CBS, and microwave irradiation.

2.3. Microwave-assisted functionalisation of rubberised aramid fabric

The rubberised aramid fabric was functionalised by microwave assistance. The microwave oven was a Samsung CE117PC with a nominal frequency of 2.45 GHz and a rated maximum microwave output power of 900 W. The specimen temperature was monitored during the intermittent microwave treatment using Infrared (IR) pyrometer, and irradiation was continued until the specimen temperature approached approximately 140 °C. Once this temperature was reached, the treatment condition was maintained for a further 2 min and the specimen was allowed to cool naturally to room temperature. Similarly, treatment temperature of approximately 140 °C was selected as a controlled processing condition intended to promote treatment-induced interfacial modification while limiting excessive thermal exposure of the aramid reinforcement. The microwave-processing parameters adopted in the present study are summarised in Table 3 [15].

The overall microwave-assisted functionalisation procedure used in

Table 3

Microwave functionalisation parameters adopted in the present study.

Parameter	Condition
Microwave oven	Samsung CE117PC
Operating frequency	2.45 GHz
Microwave output power	900 W rated maximum output*
Heating mode	Intermittent irradiation
Irradiation interval	2 min
Total irradiation time	10 min
Temperature measurement method	Infrared (IR) pyrometer
Plateau temperature	Approximately 140 °C
Holding time at/after reaching target temperature	2 min
Cooling method	Ambient air cooling

the present investigation is shown in Fig. 2 [16].

2.4. Preparation of natural rubber compound

The matrix material for the fabrication of microwave-functionalised aramid fabric composites was a natural rubber (NR) compound prepared using a conventional sulphur vulcanisation formulation. In order to achieve efficient sulphur vulcanisation, inorganic and organic cure activators were added in the form of zinc oxide and stearic acid respectively. TQ was used as an antioxidant to enhance the oxidative stability of rubber compound during processing and curing process. In addition, N660 GPF carbon black was added at a loading of 40 phr for mechanical reinforcement. The vulcanisation system consisted of sulphur as the curing agent, TBBS as the main accelerator and DPG and PVI were added to control the curing characteristics and improve the scorch safety. Table 4 gives the formulation of the natural rubber compound used in the present investigation [17].

The rubber compound was prepared on a laboratory two-roll mill according to conventional rubber compounding practice to ensure uniform dispersion of all compounding ingredients before composite fabrication. The compounded natural rubber was used as matrix material for the microwave-functionalised aramid fabric composites

Table 4

Formulation of the natural rubber compound.

Component	Loading (phr)
Natural Rubber (NR)	100
Zinc oxide	5
Stearic acid	2
TQ	2
N660 GPF carbon black	40
TBBS	0.6
DPG	0.6
Sulphur	2.5
PVI	0.3

manufacturing. The overall preparation procedure of the natural rubber compound is schematically shown in Fig. 3 [18].

2.5. Fabrication of microwave-functionalised aramid/natural rubber composites

All mastication and mixing operations for both the EPDM functionalisation masterbatch and the natural rubber compound were carried out using a water-cooled laboratory two-roll mill (SMX-LAB-613, Santhosh-GC; roll size: 6 × 13 in.) operated at a friction ratio of 1.4:1 in accordance with ASTM D3182. The compounded materials were subsequently used for fabricating the microwave-functionalised aramid/natural rubber composite panels. The composite panels were fabricated according to the specimen configuration specified for ASTM D413 peel adhesion testing. The composite structure was prepared by placing 1.5 mm thick natural rubber sheets on both sides of the microwave-functionalised rubberised aramid fabric measuring 300 × 150 mm, thereby forming a symmetric rubber–fabric–rubber sandwich structure. The EPDM-based carrier layer associated with the treated aramid fabric was retained at the interfacial region and was not separately removed prior to compression moulding. The fabrication procedure adopted for the microwave-functionalised aramid/natural rubber composites is schematically illustrated in Fig. 4 [19].

To facilitate the subsequent peel adhesion test, approximately

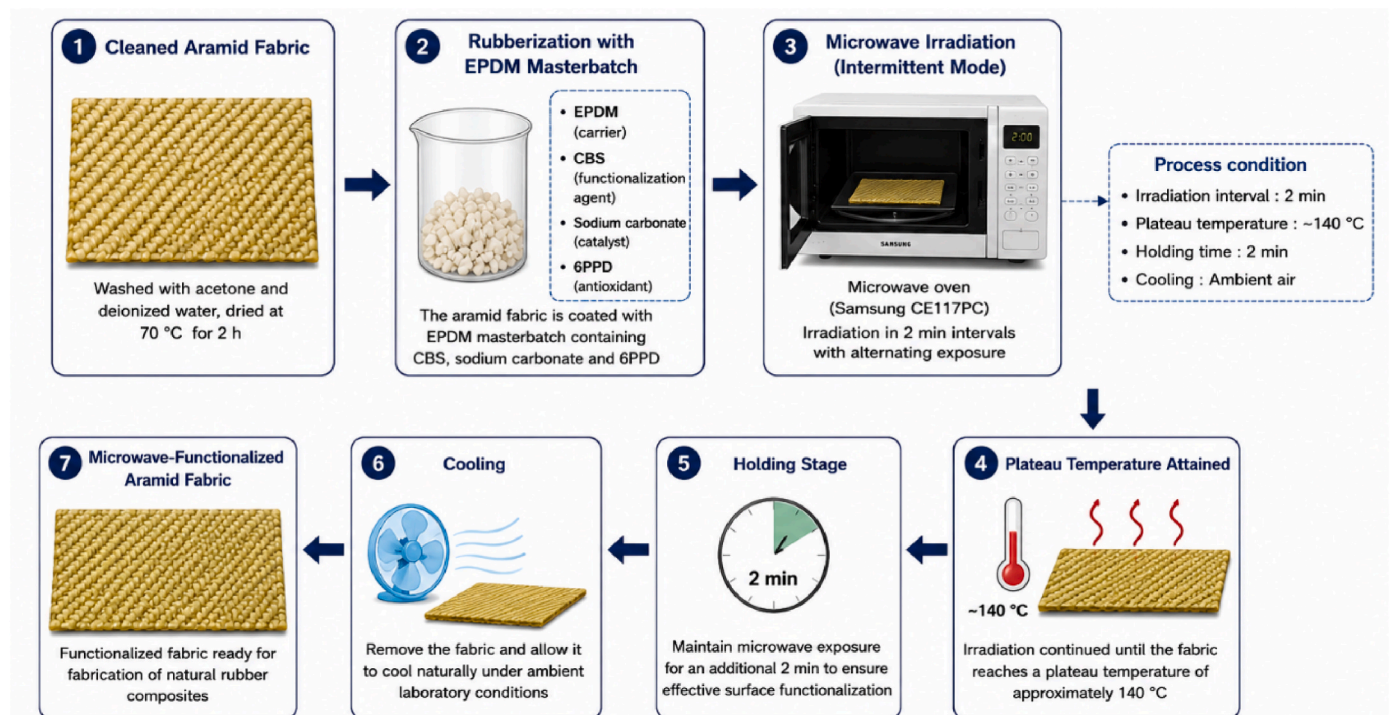


Fig. 2. Microwave-assisted functionalisation procedure for rubberised aramid fabric.

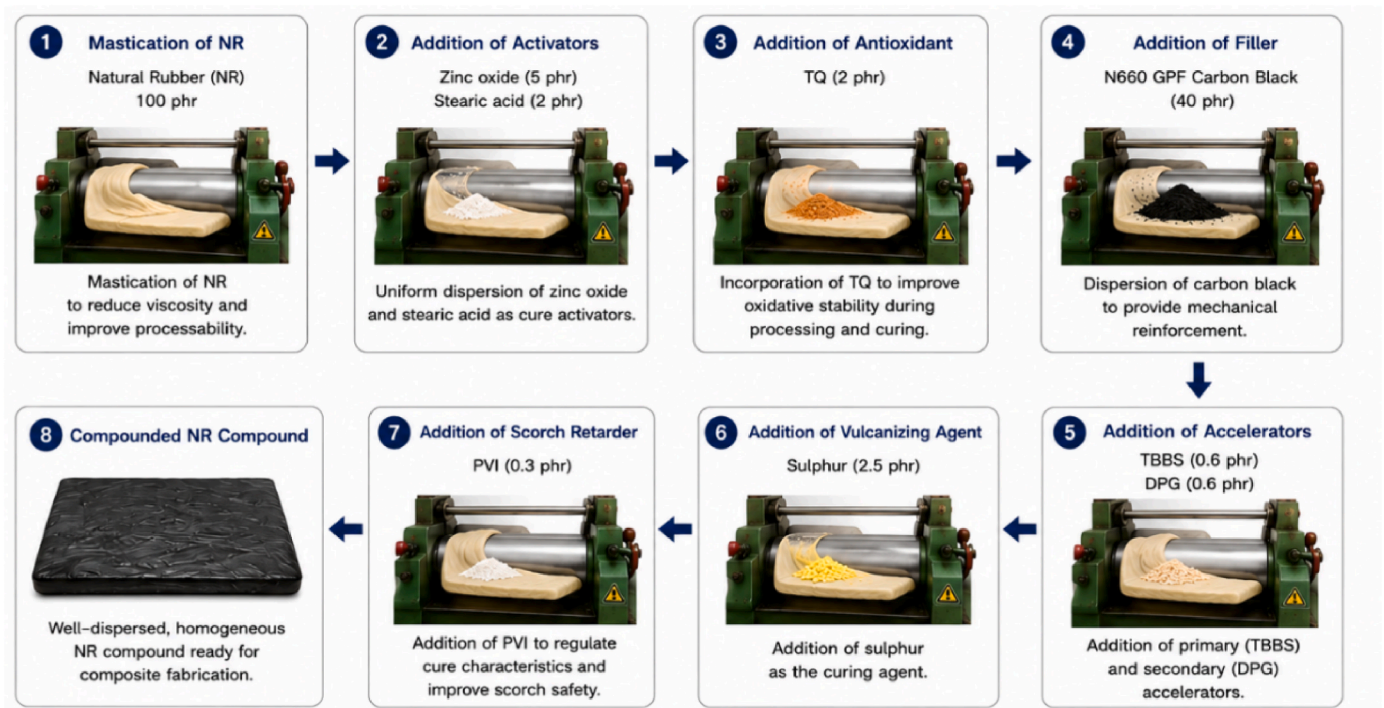


Fig. 3. Schematic representation of the preparation of the natural rubber compound using conventional two-roll mill mixing.

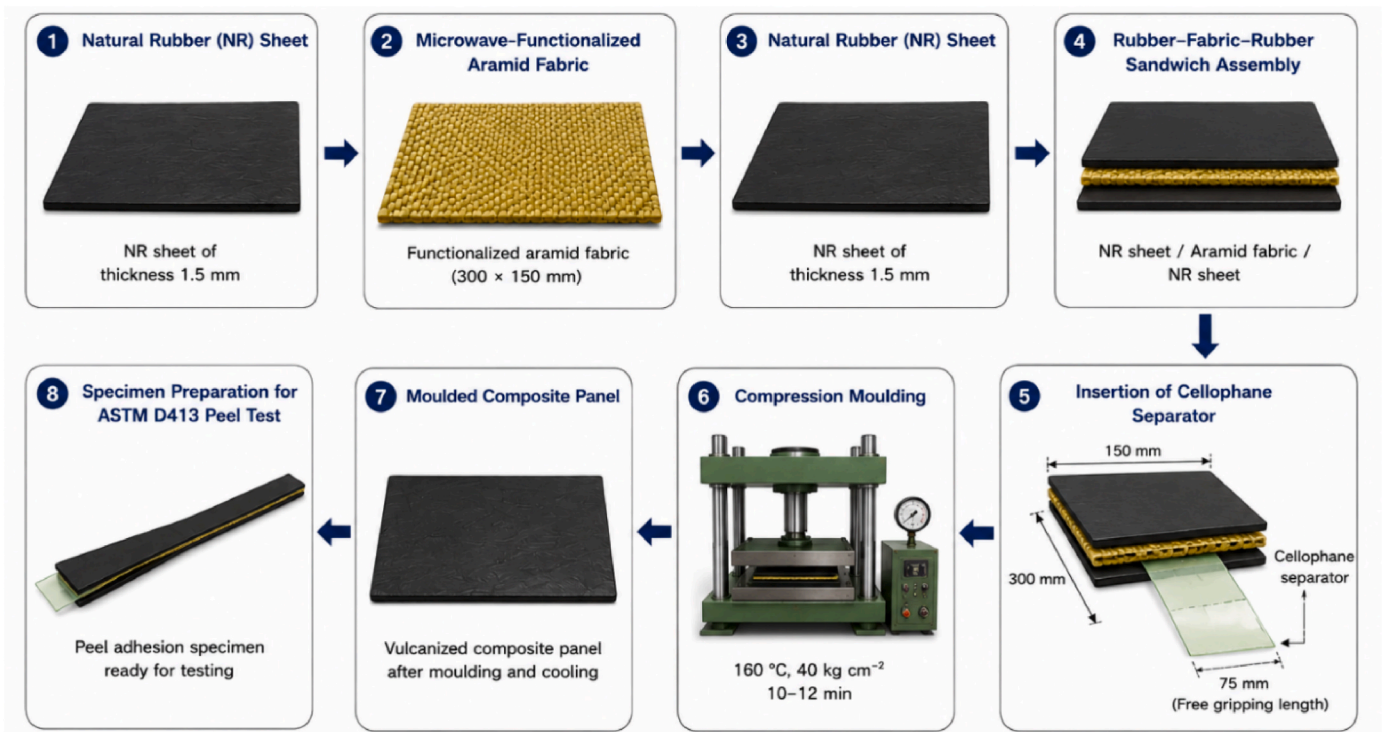


Fig. 4. Schematic representation of the fabrication procedure for microwave-functionalised aramid fabric reinforced natural rubber composite panels prior to compression moulding.

75 mm of the aramid fabric at one end of the specimen was isolated from the rubber sheet using a cellophane separator, thereby preventing bonding in this region and providing a free gripping length for tensile loading during the peel test. The prepared composite panels were compression moulded using a 150 mm daylight hydraulic compression moulding press at 160 °C for 10–12 min under a pressure of 40 kg cm⁻².

Furthermore, the selected moulding duration of 10–12 min was shorter than the rheometrically determined T_{90} of 16.81 min for the bulk NR compound; therefore, the moulding time should not be interpreted as being equivalent to the measured T_{90} . After moulding, the vulcanised composite panels were removed from the mould and test specimens of the required dimensions were prepared for peel adhesion testing. The

geometry of the prepared peel adhesion specimens is presented in Fig. 5 [20].

2.6. Peel adhesion test

The interfacial adhesion between the microwave-functionalised aramid fabric and the natural rubber matrix was evaluated using the 90° strap peel adhesion test in accordance with ASTM D413. The tests were performed using a Shimadzu AGS-2000 Universal Testing Machine (UTM) operated at a constant crosshead speed of 50 mm min⁻¹. During testing, the force required to separate the bonded rubber–fabric interface was continuously recorded throughout the peeling process. The experimental configuration adopted for the ASTM D413 peel adhesion test is schematically illustrated in Fig. 6.

During each peel test, the load–displacement response was continuously recorded and the maximum peel load attained during separation was extracted for each specimen. Three specimens were tested for each treatment condition, and the results are presented as mean ± standard deviation. Owing to the limited replicate number (n = 3), the results are interpreted descriptively, and no inferential statistical significance is claimed, among the control and E1–E5 groups. Accordingly, the comparative values reported in Fig. 12 represent the mean maximum peel load rather than the average peel force over the middle 50% of the bonded length. The specimen surfaces after peeling were visually inspected to determine the dominant mode of failure after mechanical testing. The remaining amount of rubber on the aramid fabric surface was used as the qualitative index of fibre–rubber interfacial adhesion. Fig. 7 shows representative specimens before and after peel testing [21].

2.7. Characterisation techniques

The chemical characteristics of the untreated aramid fabric and the microwave-assisted CBS/EPDM-treated rubberised fabric surface were characterised using Fourier Transform Infrared Spectroscopy (FTIR) in the spectral range of 4000–400 cm⁻¹ with a resolution of 4 cm⁻¹. Similarly, surface elemental composition and chemical states were investigated by X-ray Photoelectron Spectroscopy (XPS) using a monochromatic Al K α X-ray source (1486.6 eV). As well, Thermogravimetric Analysis (TGA) was performed under a nitrogen atmosphere from room temperature to 800°C at a heating rate of 10°C min⁻¹ to evaluate the thermal stability of the untreated and functionalised aramid fabrics. Furthermore, surface morphology and fracture surfaces after peel testing

were examined using Scanning Electron Microscopy (SEM) operated at an accelerating voltage of 10–15 kV. Lastly, obtained FTIR, XPS, TGA, and SEM results were correlated with the peel adhesion behaviour to understand the influence of the combined microwave-assisted CBS/EPDM treatment on the fibre–rubber interface [22].

3. Results and discussion

3.1. Characterisation of microwave-assisted CBS/EPDM-treated aramid fabric

The untreated aramid fabric and the E4 treatment condition were selected for detailed surface and physicochemical Characterisation because they represent the baseline and the treatment condition exhibiting the highest peel adhesion performance, respectively. For the E4 specimen, the Characterisation results represent the combined microwave-assisted CBS/EPDM treatment layer associated with the rubberised fabric surface, since the EPDM carrier was retained after treatment and was not separately removed. This comparison was intended to identify the principal surface chemical, thermal and morphological differences associated with the best-performing treatment condition. The E1–E5 series was primarily employed to establish the effect of CBS loading on peel adhesion performance, whereas detailed FTIR, XPS, TGA and SEM Characterisation was focused on the untreated and E4 specimens. Consequently, the present Characterisation does not establish a concentration-dependent evolution of the surface chemical features across all CBS loadings, and this aspect warrants systematic investigation in future work.

3.1.1. Cure characteristics of the natural rubber compound

The curing characteristics of the developed natural rubber compound were evaluated before composite fabrication to determine its processing behaviour and vulcanisation characteristics. The scorch time (TS₂) is the time at which vulcanisation starts and is an important indicator of processing safety and the optimum cure time (T₉₀) is the time at which approximately 90% of the maximum state of cure is achieved. These parameters provide a reference for understanding the processing and vulcanisation behaviour of the NR compound. The measured cure characteristics are shown in Table 5.

The natural rubber compound developed showed a longer scorch time (TS₂ = 7.08 min) than the gum natural rubber (5.60 min), indicating better processing safety in mixing and compression moulding.

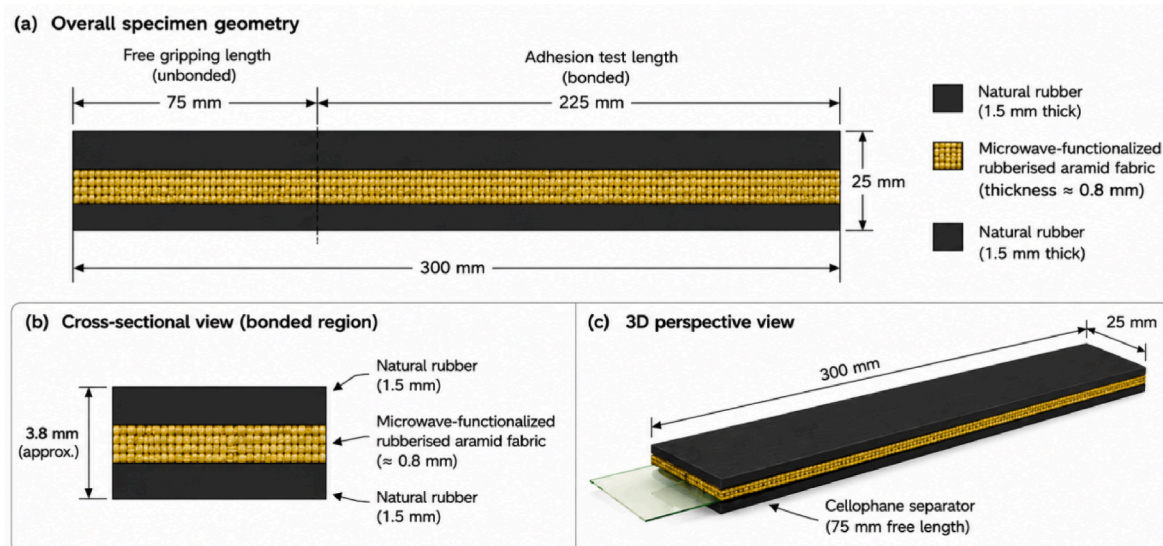


Fig. 5. Schematic representation of the aramid fabric/NR composite configuration, comprising a microwave-functionalised rubberised aramid fabric layer (0.8 mm) positioned between two NR layers (1.5 mm each), giving an approximate total thickness of 3.8 mm.

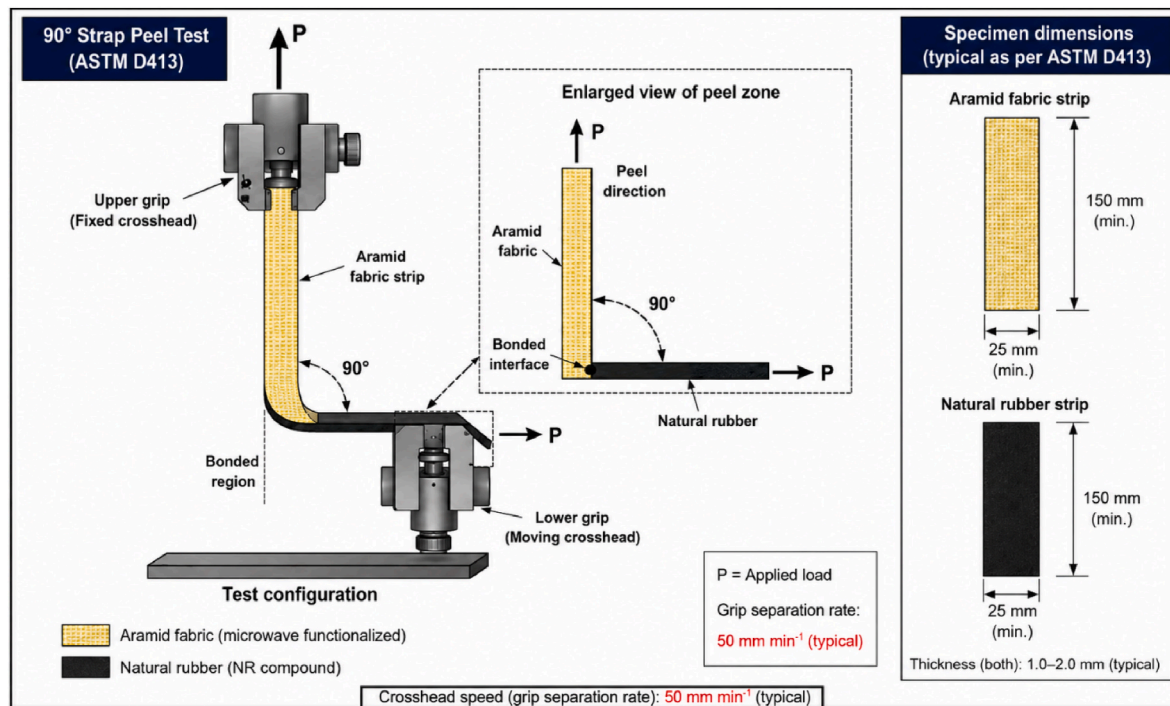


Fig. 6. Schematic illustration of the ASTM D413 90° strap peel-test configuration used to evaluate the interfacial adhesion between microwave-functionalised aramid fabric and the natural rubber matrix at a crosshead speed of 50 mm min⁻¹.

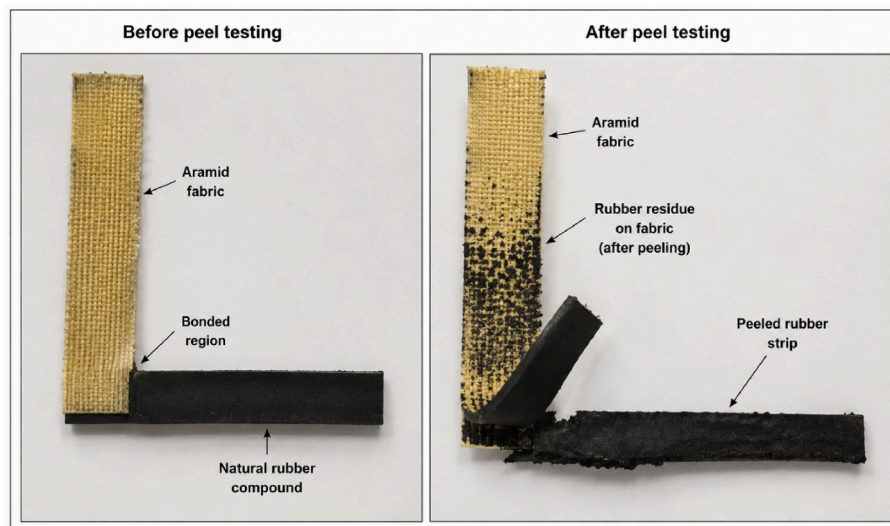


Fig. 7. Representative peel adhesion test specimens before and after ASTM D413 testing, illustrating the fracture surfaces used for qualitative failure analysis.

Table 5
Cure characteristics of the natural rubber compound.

Parameter	Gum NR	NR Compound
TS ₂ (min)	5.60	7.08
T ₉₀ (min)	11.30	16.81
T ₉₀ -TS ₂ (min)	5.70	9.73

Similarly, the optimum cure time (T₉₀) was increased from 11.30 min for the gum rubber to 16.81 min for the compounded formulation. The corresponding increase in the cure interval (T₉₀-TS₂) from 5.70 min to 9.73 min further indicates a more controlled vulcanisation process. These observations are consistent with the incorporation of the

conventional sulphur vulcanisation system, particularly the presence of PVI and DPG, which control the onset and progression of cure. An important benefit of the extended processing window for the present work was the time for the build-up of the rubber-fabric-rubber sandwich structure and subsequent compression moulding without negative impact on the curing process. It should be noted that the rheometrically determined T₉₀ of the NR compound (16.81 min) was longer than the 10–12 min compression-moulding duration used for composite fabrication. Therefore, the selected moulding time is not considered equivalent to T₉₀ and the present cure data do not establish that 90% of the maximum rheometric cure state was attained during composite moulding. The 10–12 min moulding condition represents the processing condition employed consistently for all control and treated composite

specimens in the present investigation. Accordingly, the cure characteristics are reported primarily to describe the processing behaviour of the NR compound, while the comparison of adhesion performance is restricted to specimens fabricated under the same moulding conditions [23].

3.1.2. FTIR analysis of microwave-assisted CBS/EPDM-treated aramid fabric

Fourier Transform Infrared (FTIR) spectroscopy was used to investigate the chemical characteristics of the treated/rubberised fabric surface in order to evaluate the effect of the combined microwave-assisted CBS/EPDM treatment. Fig. 8 shows the FTIR spectra of the untreated aramid fabric and the microwave-assisted CBS/EPDM-treated specimen (E4). The aim of this analysis was to check whether the microwave-assisted CBS treatment induced evident changes in the surface chemistry of the aramid fabric, while maintaining its intrinsic molecular structure. The FTIR spectrum of the untreated aramid fabric showed the typical absorption bands of aromatic polyamide (Kevlar), confirming the chemical integrity of the reinforcement. The wide absorption band around 3300 cm^{-1} was assigned to the N–H stretching vibration of amide groups. The peaks at 1640 cm^{-1} and 1540 cm^{-1} were assigned to the amide I (C=O stretching) and amide II (N–H bending and C–N stretching) vibrations. Absorption bands in the region $1500\text{--}1600\text{ cm}^{-1}$ are attributed to aromatic C=C skeletal vibrations. The bands observed between 1300 and 1400 cm^{-1} are due to C–N stretching and aromatic ring deformation typical of para-oriented aramid fibres. These spectral features are consistent with the reported molecular structure of Kevlar fibres and suggest that the untreated reinforcement had the expected chemical composition. The microwave-assisted CBS functionalisation did not significantly change the overall spectral profile of the aramid fabric, indicating that the treatment did not affect the fundamental molecular structure of the fibre. However, small changes in the intensity and shape of several characteristic absorption bands have been detected, especially in the amide and aromatic vibration regions. Also weak absorption features related to the benzothiazole structure of CBS were seen, indicating treatment-derived species associated with the treated/rubberised fabric surface. These spectral changes are consistent with changes in the chemical environment associated with the combined microwave-assisted CBS/EPDM treatment layer rather than degradation

of the aramid fibre [24]. The preservation of the characteristic Kevlar absorption bands together with the appearance of subtle spectral variations indicates changes associated with the treated/rubberised fabric surface while suggesting preservation of the characteristic aramid structure. Such surface modification is expected to enhance the interaction between the aramid fabric and the natural rubber matrix during vulcanisation, thereby contributing to improved interfacial compatibility and stress transfer. The spectral differences between the untreated and E4 specimens were relatively subtle and therefore should not be interpreted as direct evidence for the formation of a specific chemical bond between CBS-derived species and the aramid substrate or as Characterisation of the isolated bare aramid surface, because the EPDM-based carrier remained associated with the treated fabric. Rather, the observed variations provide supporting evidence of changes in the surface chemical environment following microwave-assisted treatment. Accordingly, the FTIR results are interpreted as being consistent with the presence or association of treatment-derived species at the aramid surface while preserving the characteristic molecular structure of Kevlar. These findings are considered together with the complementary XPS results presented in the subsequent section and the observed peel-test response. Thus, FTIR is used here as supporting evidence of surface modification and not as proof of a specific CBS–aramid covalent bonding mechanism [25].

3.1.3. XPS analysis of microwave-functionalised aramid fabric

X-ray Photoelectron Spectroscopy (XPS) was employed to investigate the surface elemental composition and chemical states of the untreated aramid fabric and the microwave-assisted CBS-functionalised aramid fabric (E4). Owing to its high surface sensitivity, XPS provides information on changes in the elemental composition and chemical-state environment of the outermost fibre surface following microwave-assisted treatment. The survey spectra and high-resolution core-level spectra are presented in Fig. 9.

The survey spectrum of the untreated aramid fabric (Fig. 9a) exhibits prominent carbon (C 1s), nitrogen (N 1s), and oxygen (O 1s) signals characteristic of the aromatic polyamide structure. Following microwave-assisted CBS/EPDM treatment (Fig. 9b), the C 1s, N 1s, and O 1s signals remain evident, together with an additional S 2p signal. The detection of sulphur in the treated specimen indicates the presence of

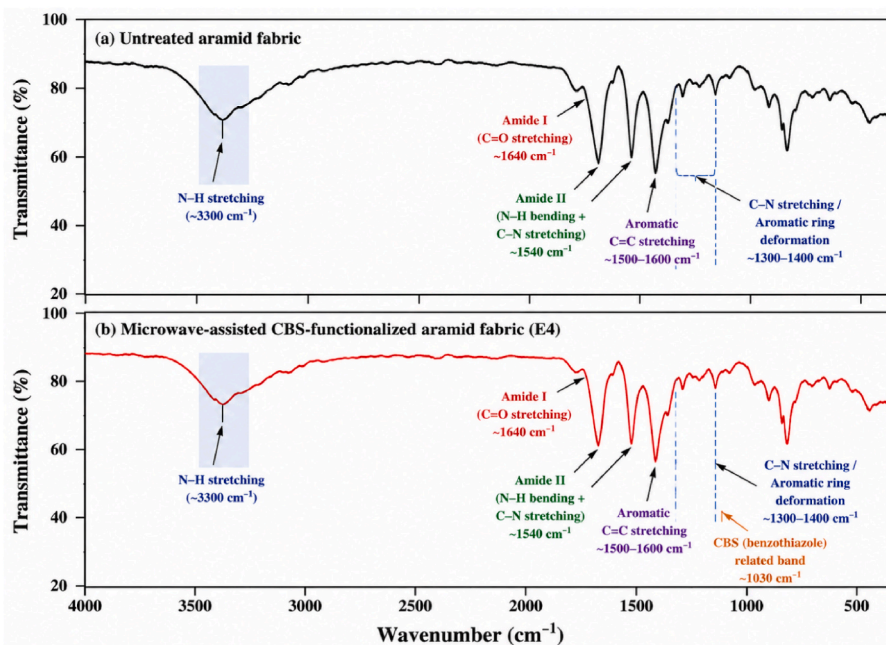


Fig. 8. FTIR spectra of untreated aramid fabric and microwave-assisted CBS-treated aramid fabric (E4), showing the characteristic absorption bands of Kevlar and subtle spectral variations following treatment.

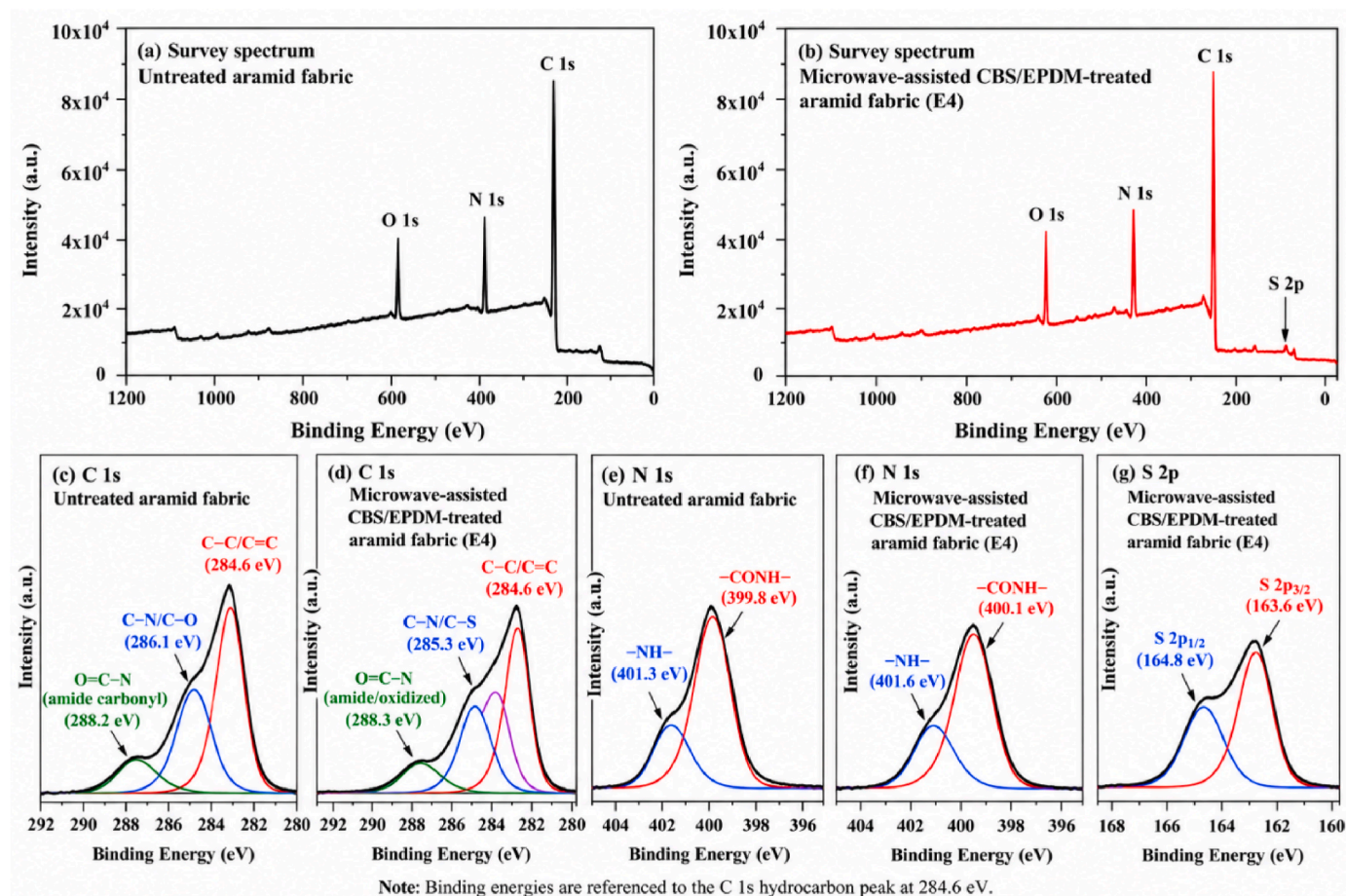


Fig. 9. X-ray Photoelectron Spectroscopy (XPS) analysis of untreated and microwave-assisted CBS-functionalised aramid fabric (E4): (a) survey spectrum of untreated aramid fabric, (b) survey spectrum of microwave-assisted CBS-functionalised aramid fabric (E4), (c) high-resolution C 1s spectrum of untreated aramid fabric, (d) high-resolution C 1s spectrum of microwave-assisted CBS-functionalised aramid fabric (E4), (e) high-resolution N 1s spectrum of untreated aramid fabric, (f) high-resolution N 1s spectrum of microwave-assisted CBS-functionalised aramid fabric (E4), and (g) high-resolution S 2p spectrum of the microwave-assisted CBS-functionalised aramid fabric, indicating the presence of sulphur-containing CBS-associated species on the fibre surface.

sulphur-containing treatment-derived species associated with the treated/rubberised fabric surface. Since the EPDM-based carrier was retained after microwave treatment, the elemental composition of the E4 specimen is interpreted as representing the combined interfacial treatment layer rather than the isolated bare aramid fibre surface. The high-resolution C 1s spectra of the untreated and treated specimens are presented in Fig. 9c and d, respectively. For the untreated aramid fabric, the C 1s spectrum was resolved into components centred at approximately 284.6, 286.1, and 288.2 eV. The component at 284.6 eV is assigned to aromatic/aliphatic C-C/C=C environments, while the component at 286.1 eV is associated with C-N/C-O environments. The higher-binding-energy component at approximately 288.2 eV is attributed to the amide carbonyl (O=C-N) environment of the aromatic polyamide structure. Following microwave-assisted CBS/EPDM treatment, changes are observed in the C 1s envelope and the relative contributions of its fitted components. The treated specimen exhibits a principal C-C/C=C contribution near 284.6 eV and an overlapping C-N/C-S contribution near 285.3 eV, together with a higher-binding-energy contribution near 288.3 eV. Considering the retained EPDM/CBS-containing treatment layer and the possible overlap of chemical environments, the higher-binding-energy contribution in the treated specimen is interpreted conservatively and is not used as evidence of a specific new chemical bond between CBS and the underlying aramid substrate. The high-resolution N 1s spectra (Fig. 9e and f) show nitrogen-containing components associated with the polyamide structure. The untreated specimen exhibits fitted contributions near 399.8 and

401.3 eV, while the corresponding components in the treated specimen occur near 400.1 and 401.6 eV. The relatively small changes in binding energy and spectral contribution following treatment indicate an alteration in the local surface chemical environment. However, because nitrogen-containing species from the treatment system may contribute to the E4 spectrum, these variations are not interpreted as direct evidence of a specific covalent interaction between CBS and the aramid substrate. The high-resolution S 2p spectrum of the treated specimen (Fig. 9g) exhibits a spin-orbit doublet with components at approximately 163.6 and 164.8 eV, assigned to S 2p_{3/2} and S 2p_{1/2}, respectively. The appearance of this sulphur-containing contribution in the treated specimen supports the presence of sulphur-containing treatment-derived species associated with the microwave-assisted CBS/EPDM treatment system. Importantly, because the EPDM-based carrier remained associated with the treated fabric, the S 2p signal is attributed to the treated/rubberised fabric surface and retained interfacial treatment layer rather than being interpreted as direct evidence of CBS chemically bonded to the bare aramid fibre. Overall, the XPS results complement the FTIR observations by demonstrating changes in the elemental composition and chemical-state environment of the treated/rubberised fabric surface. The C 1s and N 1s spectral variations, together with the appearance of the S 2p signal, support the presence of treatment-derived species and an altered surface chemical environment following the combined microwave-assisted CBS/EPDM treatment. Nevertheless, the XPS results do not establish a specific covalent bonding mechanism between these treatment-derived species and the

underlying aramid substrate. Accordingly, the XPS findings are interpreted together with the FTIR, morphological, and peel adhesion observations when evaluating the interfacial behaviour of the treated composites [26,27].

3.1.4. Thermogravimetric Analysis (TGA) of microwave-functionalised aramid fabric

Thermogravimetric Analysis (TGA) was performed to evaluate the effect of microwave-assisted CBS functionalisation on the thermal stability of the aramid fabric. The thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of the untreated aramid fabric and the microwave-assisted CBS-functionalised aramid fabric (E4) are presented in Fig. 10.

The objective of this analysis was to determine whether the microwave-assisted surface treatment influenced the thermal degradation behaviour of the aramid fibres while introducing CBS-derived surface species. The TG curves demonstrate that both untreated and microwave-assisted CBS-functionalised aramid fabrics exhibit excellent thermal stability over the investigated temperature range. Negligible weight loss was observed below approximately 150°C, indicating the absence of significant moisture or volatile constituents. This behaviour confirms that the microwave-assisted treatment did not introduce unstable low-molecular-weight species that could adversely affect the processing or service performance of the reinforcement. The onset degradation temperature (T_{onset}) of the untreated aramid fabric was observed at approximately 480–490°C, which is characteristic of aromatic polyamide fibres possessing highly ordered molecular chains and extensive intermolecular hydrogen bonding. The microwave-assisted CBS-functionalised specimen exhibited a comparable onset degradation temperature with only a marginal reduction, suggesting that the functionalisation process did not significantly influence the inherent thermal resistance of the Kevlar fibres. The slight difference in the initial degradation behaviour may be associated with the thermal response of treatment-derived constituents present within the retained CBS/EPDM interfacial treatment layer. The DTG curves further reveal that both untreated and microwave-functionalised specimens exhibited a single dominant degradation peak corresponding to the decomposition of the

aromatic polyamide backbone. The maximum degradation temperature (T_{max}) remained nearly unchanged for the treated specimen compared with the untreated aramid fabric, indicating that the combined treatment did not substantially alter the principal high-temperature degradation behaviour observed for the aramid-containing treated fabric. The preservation of the principal degradation peak demonstrates that the treatment conditions were sufficiently mild to avoid thermal damage to the reinforcement. Furthermore, both specimens retained a substantial amount of residual char at temperatures between 700 and 800°C, reflecting the inherent thermal stability and carbonaceous residue formation characteristic of Kevlar fibres. The microwave-assisted CBS-functionalised specimen exhibited a residual mass comparable to that of the untreated fabric, indicating that the surface modification had no detrimental effect on the high-temperature stability of the reinforcement. Any slight variation in the residual char yield can reasonably be attributed to the presence of sulphur-containing CBS-derived surface species rather than degradation of the fibre itself. In general, the TG and DTG analyses demonstrate that microwave-assisted CBS functionalisation successfully modified the surface of the aramid fabric while preserving its excellent thermal stability. The comparable T_{onset} and T_{max} values of the untreated and microwave-assisted CBS-treated aramid fabrics indicate that the treatment did not substantially alter the overall thermal degradation behaviour of the aramid reinforcement. The small differences observed after treatment may be associated with the presence of treatment-derived species; however, the overall thermal stability of the aramid fabric was largely preserved. It should be emphasised that TGA provides information on bulk thermal degradation behaviour and does not directly determine the depth or spatial distribution of the surface treatment. Therefore, the similarity in T_{onset} and T_{max} is interpreted as evidence of largely preserved bulk thermal stability rather than proof that the treatment was confined exclusively to the fibre surface [28].

3.1.5. Scanning Electron Microscopy (SEM) analysis of microwave-functionalised aramid fabric

Scanning Electron Microscopy (SEM) was employed to examine the surface morphology of the untreated aramid fabric and the microwave-

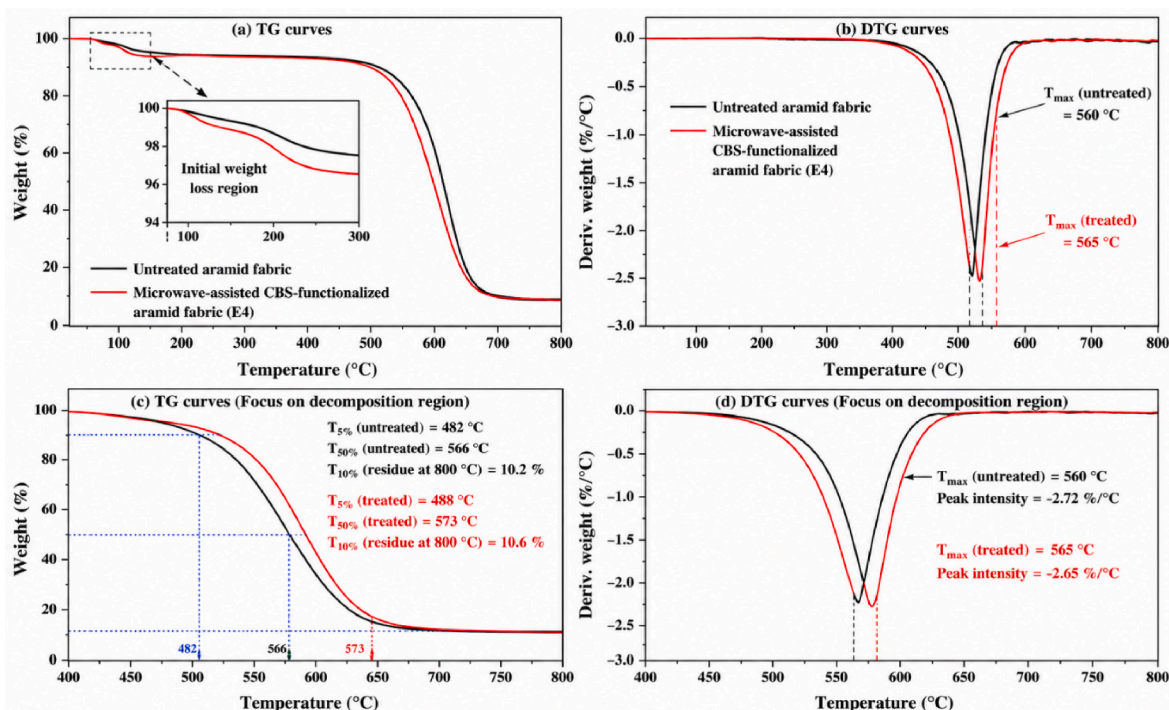


Fig. 10. TGA curves of untreated and microwave-assisted CBS-treated aramid fabric (E4), showing comparable thermal degradation behaviour following treatment.

assisted CBS-functionalised aramid fabric (E4), as well as the fracture surfaces of the corresponding peel-tested specimens. The SEM micrographs presented in Fig. 11 provide direct visual evidence of the morphological changes induced by microwave-assisted CBS functionalisation and their influence on the fibre–rubber interfacial adhesion.

The untreated aramid fabric (Fig. 11a) exhibits a relatively smooth and uniform fibre surface with well-defined longitudinal fibrillar features, which are characteristic of Kevlar fibres. The absence of noticeable surface deposits or irregularities indicates the chemically inert nature of the untreated fibre surface. Such a smooth morphology generally provides limited mechanical interlocking with the surrounding rubber matrix, resulting in comparatively weaker interfacial adhesion. Following microwave-assisted CBS functionalisation, the surface morphology of the aramid fibres changed noticeably (Fig. 11b). The treated fibres exhibited a relatively rougher surface with localised surface irregularities distributed along the fibre surface. These morphological changes are consistent with the presence of the retained treatment layer and treatment-derived species associated with the treated/rubberised fabric surface, as supported by the FTIR and XPS analyses. The increased surface roughness is expected to improve the effective contact area between the aramid fibres and the natural rubber matrix, thereby enhancing mechanical interlocking and promoting stronger interfacial interaction during vulcanisation. The fracture surface of the untreated control specimen after the peel adhesion test (Fig. 11c) reveals a predominantly clean fibre surface with minimal rubber residue, indicating that interfacial debonding occurred primarily at the fibre–rubber interface. This adhesive failure mode confirms the relatively poor interfacial compatibility of the untreated aramid fabric with the natural rubber matrix and is consistent with the lower peel adhesion strength measured for the control specimen. In contrast, the fracture surface of the microwave-assisted CBS-functionalised specimen (E4) shown in Fig. 11d exhibits substantial rubber remnants firmly adhered to the fibre surface together with evidence of torn rubber fragments surrounding the fibres [29]. This mixed adhesive–cohesive failure mode indicates that the interfacial bond strength exceeded the

cohesive strength of portions of the rubber matrix, resulting in partial tearing of the rubber during peeling. The presence of retained rubber on the treated fibre surface provides clear morphological evidence of improved fibre–rubber interaction and agrees well with the significantly higher peel adhesion strength observed for the E4 composite. From the entire observations, it was found that, the SEM observation provides balancing morphological evidence supporting the FTIR, XPS, and TGA analyses. The combined Characterisation indicates that the microwave-assisted CBS/EPDM treatment altered the characteristics of the treated/rubberised fabric surface and interfacial region, resulting in increased surface roughness, improved fibre–rubber compatibility, and enhanced interfacial adhesion, resulting in increased surface roughness, improved fibre–rubber compatibility, and enhanced interfacial adhesion. The excellent agreement between the surface Characterisation techniques and the peel adhesion results confirms that microwave-assisted CBS functionalisation is an effective and practical surface engineering strategy for improving the performance of aramid fabric–reinforced natural rubber composites. The improved surface morphology observed after microwave-assisted CBS functionalisation is expected to facilitate efficient stress transfer across the fibre–rubber interface, thereby contributing to the superior peel adhesion performance of the E4 composite [30].

3.2. Peel adhesion performance

The interfacial adhesion between microwave functionalised aramid fabric and natural rubber matrix was quantified by the ASTM D413 90° strap peel adhesion test. A control specimen of an untreated aramid fabric reinforced composite was used for the baseline adhesion behaviour. Specimens E1–E5 were prepared with increasing concentrations of CBS in the microwave-assisted functionalisation process. Table 6 summarises the maximum peel load and the maximum displacement measured for each specimen. For comparative evaluation, the maximum peel-load values obtained from the three replicate specimens of each treatment condition were averaged, and these mean maximum peel-load

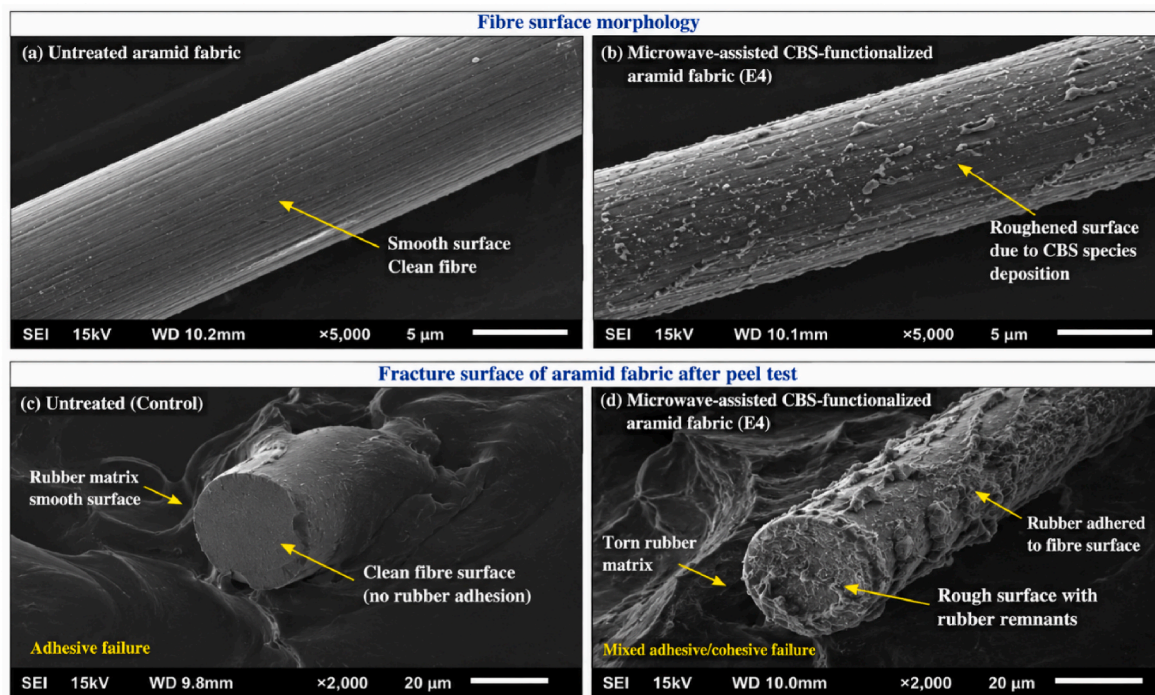


Fig. 11. Scanning Electron Microscopy (SEM) images of untreated and microwave-assisted CBS-functionalised aramid fabric (E4): (a) untreated aramid fibre surface, (b) microwave-assisted CBS-functionalised aramid fibre surface (E4), (c) fracture surface of the untreated control specimen after the ASTM D413 peel adhesion test showing adhesive failure with minimal rubber residue, and (d) fracture surface of the microwave-assisted CBS-functionalised specimen (E4) after the peel adhesion test showing mixed adhesive–cohesive failure with retained rubber fragments on the fibre surface, indicating enhanced interfacial adhesion.

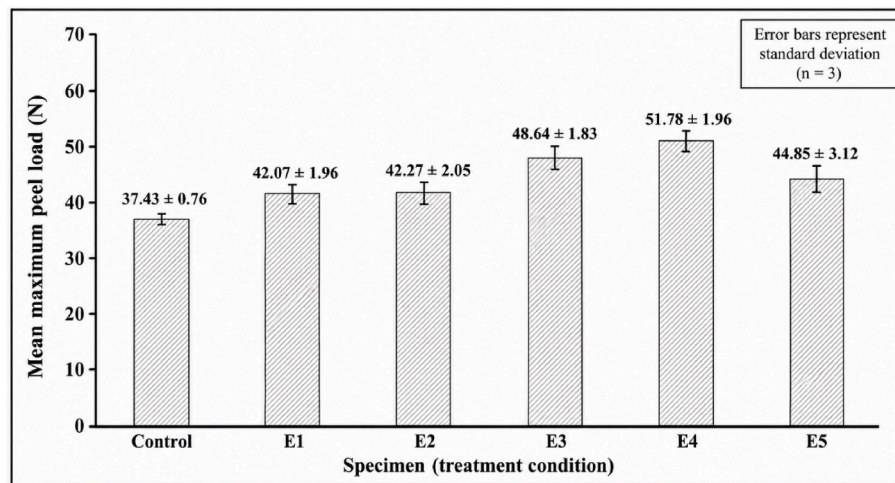


Fig. 12. Mean maximum peel load of the control and microwave-functionalised aramid/NR composite specimens for the E1–E5 treatment conditions. Values are presented as mean $\pm$ standard deviation ($n = 3$).

Table 6

Maximum peel-load and corresponding displacement values recorded for the control and microwave-functionalised aramid/NR composite specimens.

Specimen	Test No.	Maximum peel load (N)	Maximum displacement (mm)
Control	1	37.27	648.4
	2	36.77	623.7
	3	38.25	635.2
E1	1	44.23	634.2
	2	41.38	641.8
	3	40.60	631.9
E2	1	40.40	643.5
	2	44.42	639.1
	3	41.97	641.2
E3	1	47.56	623.7
	2	48.25	638.2
	3	50.11	642.5
E4	1	51.09	621.7
	2	54.03	631.5
	3	50.21	628.1
E5	1	48.15	618.2
	2	42.17	624.8
	3	44.23	617.6

values were used in Fig. 12.

The experimental results indicate that the treated specimens exhibited higher mean maximum peel loads than the untreated control under the investigated conditions. The mean maximum peel load of 37.43 N was observed for the untreated control specimen which could be attributed to weak interfacial interaction between the chemically inert aramid surface and non-polar natural rubber matrix. The microwave-assisted functionalisation resulted in all treated specimens having higher peel strengths than the control, demonstrating the effectiveness of the proposed surface modification strategy. The mean maximum peel load increased from 42.07 N for E1 and 42.27 N for E2 to 48.64 N for E3 and reached the highest value of 51.78 N for E4, corresponding to an improvement of approximately 38.3% relative to the untreated control. The mean maximum peel load was nearly unchanged between E1 and E2, increased for E3, and reached its highest value for E4, after which a clear decrease was observed for E5. The observed improvement is in agreement with an increased interfacial compatibility between the treated aramid fabric and the natural rubber matrix which may have facilitated a more efficient stress transfer across the bonded interface during peel loading [31,32]. This leads to a higher resistance to the interfacial debonding and thus higher peel adhesion strengths. Similarly, further increase of CBS concentration (E5) led to the decrease of the average peel adhesion strength to 44.82 N, but this value was higher

than the untreated control. This behaviour suggests that an excessive concentration of CBS did not provide additional adhesion benefits. The reduction in peel adhesion observed for E5 may be associated with changes in the local interfacial environment at the higher CBS loading. Possible mechanisms include alterations in local vulcanisation behaviour, non-uniform distribution of treatment-derived species, or reduced effectiveness of the interfacial treatment at excessive CBS loading. However, these mechanisms were not directly evaluated in the present study and should therefore be regarded as plausible interpretations rather than experimentally confirmed explanations. These observations demonstrate that the peel adhesion response of the combined microwave-assisted CBS/EPDM treatment system was dependent on the investigated CBS loading. Among the investigated formulations, with E4 exhibiting the highest mean maximum peel load among the treatment conditions investigated in the present study. The variation in the average peel adhesion strength of the control and microwave-functionalised composite specimens is presented in Fig. 12 [33,34].

Peel adhesion increased with the treatment condition up to E4, while detailed Characterisation of the E4 specimen provided evidence of surface modification relative to the untreated aramid fabric. Because detailed surface Characterisation was focused on the untreated and E4 specimens, a concentration-dependent evolution of surface chemistry across the complete E1–E5 series cannot be established from the present data. Therefore, the relationship between CBS loading and the extent of surface modification should be interpreted cautiously. Systematic Characterisation across all treatment conditions would provide further insight into the relationship between CBS concentration, surface modification, and interfacial adhesion.

3.3. Failure surface analysis

Following the ASTM D413 peel adhesion test, the fracture surfaces of all composite specimens were visually examined to identify the dominant failure mechanism at the rubber–fabric interface. The amount of residual natural rubber remaining on the aramid fabric after peeling was used as a qualitative indicator of interfacial adhesion. In general, a relatively clean fabric surface after testing indicates adhesive failure at the rubber–fabric interface, whereas increasing amounts of rubber retained on the fabric indicate a transition toward mixed adhesive-cohesive failure, reflecting improved resistance to interfacial debonding. Representative photographs of the untreated control specimen before and after peel testing are presented in Fig. 13 [35].

In the untreated control specimen, almost complete separation of the

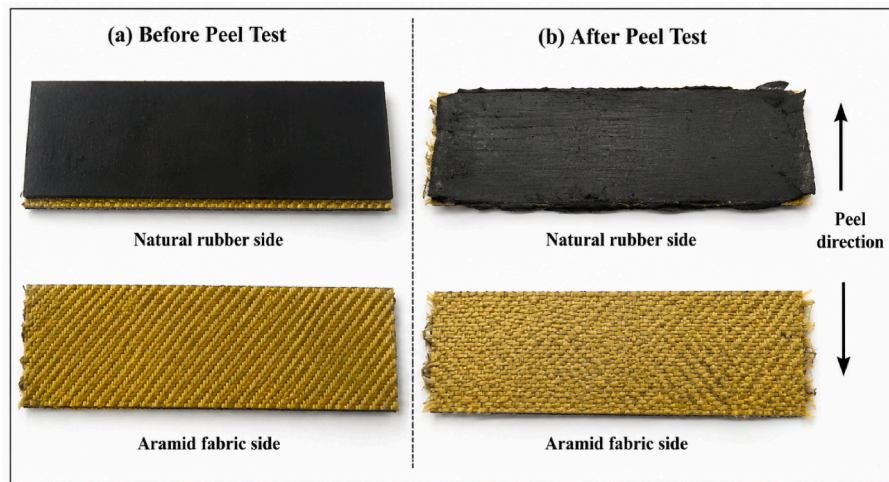
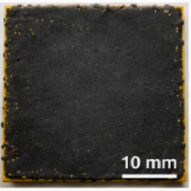
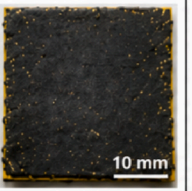
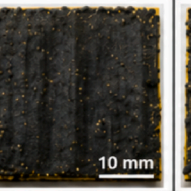
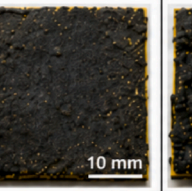
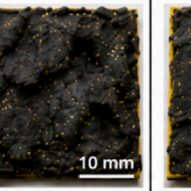
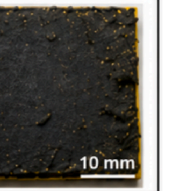
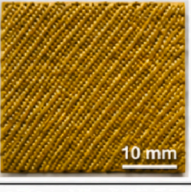
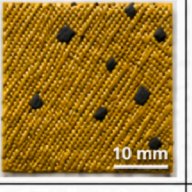
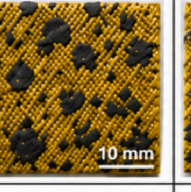
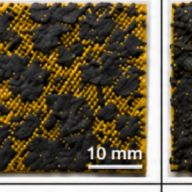
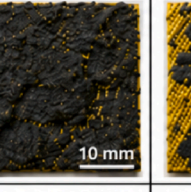
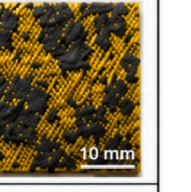


Fig. 13. Representative control specimen before and after ASTM D413 peel adhesion testing, illustrating the fracture surfaces used for qualitative failure analysis.

natural rubber from the aramid fabric was seen and only a negligible amount of rubber was left attached to the fabric surface after peeling. This type of fracture appearance indicates that the failure was primarily at the rubber–fabric interface, revealing weak interfacial adhesion between the untreated aramid fabric and the natural rubber matrix. The untreated control exhibited the lowest mean maximum peel load of 37.43 N, indicating that the failure process was governed by the interfacial debonding. On the contrary, the microwave treated specimens exhibited more and more rubber on the aramid fabric surface as the concentration of CBS increased from E1 to E4 [36]. The increase in rubber retention implies that the interfacial bond was strong enough that portions of the rubber matrix failed in peeling rather than failure being limited to the rubber–fabric interface. The combined increase in rubber retention on the aramid surface and the overall increase in mean maximum peel load from the untreated control to E4 are consistent with

a transition from predominantly interfacial adhesive failure toward mixed adhesive–cohesive failure. The amount of residual rubber left on the fabric surface was slightly decreased for specimen E5 in comparison to specimen E4, which is in accordance with the decrease in the average peel adhesion strength from 51.78 N to 44.82 N E5 still showed better adhesion than the untreated control specimen. However, the fracture morphology shows that increasing the CBS concentration above the optimum level did not lead to further interfacial toughening. Among the investigated formulations, E4 exhibited the highest mean maximum peel load and was therefore identified as the best-performing treatment condition within the experimental range considered in the present study. The comparative fracture appearances of all composite specimens are shown in Fig. 14 [37–39].

	Untreated control (no EPDM/CBS treatment; no microwave irradiation)	E1 (5.6 g CBS per 100 g EPDM)	E2 (11.2 g CBS per 100 g EPDM)	E3 (16.8 g CBS per 100 g EPDM)	E4 (22.4 g CBS per 100 g EPDM)	E5 (28.0 g CBS per 100 g EPDM)
Natural rubber side (first peeled)						
Aramid fabric side (second peeled)						
	Predominantly adhesive failure; very little rubber retention on aramid fabric.	Adhesive failure with slight increase in rubber retention.	Transition from adhesive to mixed adhesive–cohesive failure.	Mixed adhesive– cohesive failure; moderate rubber retention.	Predominantly mixed adhesive– cohesive failure; maximum rubber retention.	Mixed adhesive– cohesive failure with reduction in rubber retention compared to E4.

Note: Upper row – natural rubber side (first peeled from aramid fabric); Lower row – aramid fabric side (second peeled from natural rubber).

Fig. 14. Comparison of fracture surfaces of the control and microwave-functionalised aramid/NR composite specimens after peel adhesion testing for E1–E5 treatment conditions corresponding to CBS loadings of 5.6, 11.2, 16.8, 22.4 and 28.0 g per 100 g EPDM, respectively.

3.4. Effect of microwave-assisted CBS functionalisation on interfacial adhesion

The results obtained in the present investigation demonstrate that microwave-assisted CBS functionalisation is an effective approach for enhancing the interfacial adhesion between woven aramid fabric and the natural rubber matrix. Owing to their highly oriented molecular structure and chemically stable surface, untreated aramid fibres generally exhibit limited interaction with non-polar elastomeric matrices. Consequently, the untreated control specimen displayed the lowest peel adhesion strength and predominantly adhesive failure, indicating weak stress transfer across the rubber–fabric interface. The functionalisation strategy adopted in this work utilised an EPDM-based masterbatch as the carrier phase for distributing the CBS-containing treatment system over the aramid fabric prior to microwave irradiation. The EPDM-based carrier associated with the treated fabric was retained at the interfacial region during subsequent composite fabrication. Therefore, the observed improvement in adhesion should be attributed to the combined microwave-assisted CBS/EPDM treatment system under the investigated conditions, rather than exclusively to microwave irradiation or CBS functionalisation. Since EPDM-only, microwave-only, and CBS-without-microwave control groups were not included, the individual contributions of these treatment components cannot be independently quantified from the present experimental design [40,41]. The progressive increase in peel adhesion strength from the untreated control specimen to formulation E4, together with the corresponding transition from predominantly adhesive failure to mixed adhesive–cohesive failure, provides consistent evidence that microwave-assisted CBS functionalisation improved the effectiveness of the rubber–fabric interface. The improved interfacial compatibility likely promoted more efficient stress transfer during peel loading, thereby delaying crack initiation and increasing the resistance to interfacial debonding [42,43]. The improvement in interfacial performance is further supported by the comprehensive material Characterisation carried out in the present investigation. FTIR analysis indicated subtle changes in the surface functional groups following microwave-assisted CBS treatment, while XPS confirmed the presence of sulphur-containing CBS species on the aramid fibre surface. TGA demonstrated that the functionalisation process preserved the inherent thermal stability of the aramid fibres, and SEM observations revealed increased surface roughness together with greater rubber retention after peel testing. Collectively, these complementary Characterisation techniques provide strong evidence that microwave-assisted CBS functionalisation effectively modified the treated/rubberised fabric surface and interfacial region, resulting in enhanced fibre–rubber interfacial compatibility and improved stress transfer during peel loading. A further increase in CBS concentration beyond the E4 treatment condition resulted in a measurable reduction in peel adhesion strength for specimen E5. This behaviour suggests that the beneficial influence of CBS is concentration dependent and that excessive accelerator loading does not provide additional improvements in interfacial performance. Instead, higher CBS concentrations may influence the local vulcanisation behaviour at the rubber–fabric interface, thereby reducing the effectiveness of the surface modification. However, the E5 formulation still exhibited improved adhesion relative to the untreated control specimen confirming the overall effectiveness of the proposed microwave-assisted functionalisation strategy. In the case of peel adhesion strength, the formulation E4 had the highest average value (51.78 N), which was about 38.2% higher than the value for the untreated control. The combined results of cure characteristics, peel adhesion measurements and fracture surface observations show that controlled microwave-assisted CBS functionalisation offers a practical and effective way to enhance the fibre–rubber interface without altering conventional natural rubber compounding and compression moulding processes. These results demonstrate the potential of the investigated microwave-assisted CBS/EPDM treatment system for improving the interfacial performance of aramid fabric–reinforced natural rubber

composites under the processing conditions evaluated in the present study. Although the untreated control and the CBS-loaded treatment groups clearly demonstrate the adhesion response associated with the combined microwave-assisted CBS/EPDM treatment, the present experimental design does not include EPDM-only, microwave-only, or CBS-without-microwave control groups. Consequently, the individual contributions of the EPDM carrier, microwave irradiation, and CBS cannot be quantitatively separated. The observed enhancement should therefore be interpreted as the response of the combined treatment system, with CBS concentration acting as the systematically varied parameter. Future investigations incorporating these additional control groups would enable quantitative deconvolution of the individual treatment effects [44,45].

4. Conclusions

Microwave-assisted CBS functionalisation technique was used to improve the interfacial adhesion of woven aramid fabric/natural rubber composites. In a systematic study, the effect of CBS concentration on the peel adhesion behaviour of microwave-functionalised aramid fabric was explored and it was demonstrated that controlled surface modification can substantially improve fibre–rubber compatibility without modification of conventional rubber processing procedures. The main results of the present investigation can be summarised as follows:

1. The combined microwave-assisted CBS/EPDM treatment was associated with improved interfacial compatibility between the aramid fabric and natural rubber matrix, resulting in a measurable increase in peel adhesion performance compared with the untreated control.
2. The EPDM masterbatch served as the carrier phase for distributing the CBS-containing treatment system over the aramid fabric, and no separate EPDM-removal step was performed after microwave treatment. Consequently, the EPDM-based carrier remained associated with the treated fabric and was retained at the interfacial region during final NR composite fabrication.
3. ASTM D413 peel testing showed that the mean maximum peel load was nearly unchanged between E1 and E2, increased for E3, and reached its highest value for E4 before decreasing at E5. The E4 treatment condition exhibited the highest mean maximum peel load of 51.78 N among the investigated conditions, corresponding to an improvement of approximately 38.2% relative to the untreated control.
4. TGA showed comparable thermal degradation characteristics for the untreated and treated aramid fabrics, indicating that the overall thermal stability of the reinforcement was largely preserved following treatment.
5. Visual analysis of the fracture surfaces revealed a gradual transition from predominantly adhesive failure in the untreated control specimen to a combination of adhesive–cohesive failure in the microwave-functionalised composites, thereby providing qualitative evidence for enhanced interfacial bonding.
6. Further increasing the CBS concentration from E4 to E5 resulted in a decrease in the mean maximum peel load, indicating that the interfacial response was dependent on CBS loading, with E4 exhibiting the highest mean maximum peel load among the investigated treatment conditions.

In summary, the present investigation demonstrates that the combined microwave-assisted CBS/EPDM treatment can enhance the interfacial performance of aramid fabric–natural rubber composites under the investigated laboratory conditions. The observed improvement in peel response, together with the corresponding changes in fracture-surface characteristics, supports the potential of this treatment approach for further development of fabric-reinforced rubber composites. Additional studies addressing process scale-up, long-term durability, environmental impact, and industrial processing conditions

would be required before broader practical implementation can be established.

CRediT authorship contribution statement

H. Sheik Mohammed: Writing – review & editing. **G. Suresh:** Writing – original draft, Conceptualization. **P. Chandramohan:** Investigation. **R. Vezhavendhan:** Formal analysis. **Dinesh Kumar Madheswaran:** Methodology. **R. Saranya:** Supervision. **S. Sudagar:** Visualization.

Consent to participate

Not Applicable.

Ethical approval

This article does not contain any studies with human participants performed by any of the authors.

Code availability

Not Applicable.

Consent for publication

Not Applicable.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

References

- [1] P.J. De Lange, P.G. Akker, Adhesion activation of Twaron aramid fibres for application in rubber: Plasma versus chemical treatment, *J. Adhes. Sci. Technol.* 26 (6) (2012) 827–839, <https://doi.org/10.1163/016942411X580036>.
- [2] P.J. De Lange, P.G. Akker, S. Willemsen, R.N. Datta, The effect of oily finish components on the adhesion between aramid fibres and rubber, *J. Adhes. Sci. Technol.* 23 (1) (2009) 139–149, <https://doi.org/10.1163/156856108X344568>.
- [3] Ş. Ursache, C. Cerbu, A. Hadâr, Characteristics of carbon and Kevlar fibres, their composites and structural applications in civil engineering—A review, *Polymers* 16 (1) (2024) 127, <https://doi.org/10.3390/polym16010127>.
- [4] L. Penn, F. Larsen, Physicochemical properties of Kevlar 49 fibre, *J. Appl. Polym. Sci.* 23 (1) (1979) 59–73, <https://doi.org/10.1002/app.1979.070230106>.
- [5] N. Kanbargi, A.J. Lesser, Improving adhesion between aramid fibres and natural rubber through morphological and synthetic modification of the fibres, *J. Appl. Polym. Sci.* 135 (24) (2018) 45520, <https://doi.org/10.1002/app.45520>.
- [6] M. Shirazi, M.B. de Rooij, A.G. Talma, J.W.M. Noordermeer, Adhesion of RFL-coated aramid fibres to elastomers: the role of elastomer–latex compatibility, *J. Adhes. Sci. Technol.* 27 (17) (2013) 1886–1898, <https://doi.org/10.1080/01694243.2013.764145>.
- [7] H. Şenol, H. Ulus, A. Al-Nadhari, S. Topal, M. Yıldız, Ameliorating tensile and fracture performance of carbon fibre-epoxy composites via atmospheric plasma activation: insights into damage modes through in-situ acoustic emission inspection, *Compos. Appl. Sci. Manuf.* 195 (2025) 108929, <https://doi.org/10.1016/j.compositesa.2025.108929>.
- [8] L. Liu, S. Liang, D. Wang, Y. Zhou, C. Zheng, Effect of secondary modification of silane coupling agent and graphene oxide on interfacial properties of aramid fibre, *J. Mater. Eng.* 50 (1) (2022) 145–153, <https://doi.org/10.11868/j.issn.1001-4381.2020.001116>.
- [9] L. Yin, B. Zhang, M. Tian, N. Ning, L. Zhang, W. Wang, Surface construction of ANF/CNT onto aramid fibres to enhance interfacial adhesion and provide real-time monitoring of deformation, *Compos. Sci. Technol.* 223 (2022) 109336, <https://doi.org/10.1016/j.compscitech.2022.109336>.
- [10] C. Li, Y. Cui, W. Xing, X. Pei, J. Song, L. Wang, Secondary silane grafting on aramid fibres improves the interfacial bonding performance in textile composite materials, *J. Compos. Mater.* 56 (29) (2022) 4421–4432, <https://doi.org/10.1177/00219983221129690>.
- [11] T. Xu, Z. Qi, Q. Yin, Y. Jiao, L. An, Y. Tan, Effects of air plasma modification on aramid fibre surface and its composite interface and mechanical properties, *Polymers* 22 (2022) 4892, <https://doi.org/10.3390/polym14224892>.
- [12] Z. Fang, Q. Tu, X. Yang, X. Shen, Q. Yin, Z. Chen, Polydopamine and mercapto functionalised 3D carbon nano-material hybrids synergistically modifying aramid fibres for adhesion improvement, *Polymers* (2022). <https://pubmed.ncbi.nlm.nih.gov/36235933/>.
- [13] C. Cao, J. Peng, X. Liang, E. Saiz, S.E. Wolf, H.D. Wagner, L. Jiang, Q. Cheng, Strong, conductive aramid fiber functionalized by graphene, *Compos. Appl. Sci. Manuf.* 140 (2021) 106161, <https://doi.org/10.1016/j.compositesa.2020.106161>.
- [14] N. Ramasamy, V. Arumugam, C. Suresh Kumar, Characterisation of fibre/matrix interfacial bonding strength of chemically grafted aramid fibre surface with epoxy resin composites, *Polymer Composites*, Advance online publication, 2022, <https://doi.org/10.1002/pc.26384>.
- [15] H.-O. Ruan, H. Du, T. Umemoto, R. Ono, D. Katase, A. Kumada, M. Sato, Effect of O radicals on aramid surface treatment: experimental and molecular insights, *Appl. Phys. Lett.* 125 (17) (2024) 171602, <https://doi.org/10.1063/5.0237844>.
- [16] J. Lee, Study on the interfacial stability of rubber/copolymer aramid by surface treatment of copolymer aramid nanofibre, *Polymer (Korea)* 48 (2) (2024) 158–164, <https://doi.org/10.7317/pk.2024.48.2.158>.
- [17] C.Y. Yue, K. Padmanabhan, Interfacial studies on surface modified Kevlar fibre/epoxy matrix composites, *Compos. B Eng.* 30 (2) (1999) 205–217, [https://doi.org/10.1016/S1359-8368\(98\)00053-5](https://doi.org/10.1016/S1359-8368(98)00053-5).
- [18] W. Xu, Y. Song, J. Ye, X. Li, T. Qiu, X. Tuo, Surface decoration and functionalization on polymerization-induced aramid nanofibres: implications for barrier films and light-to-heat conversion, *ACS Appl. Nano Mater.* 5 (8) (2022) 11059–11070, <https://doi.org/10.1021/acsnm.2c02252>.
- [19] J. Wang, X. Zhang, Y. Wen, Y. Chen, Q. Fu, J. Wang, H. Jia, Aramid nanofibre/XNBR nanocomposite with high mechanical, thermal, and electrical performance, *Nanomaterials* 13 (2) (2023) 335, <https://doi.org/10.3390/nano13020335>.
- [20] Z. Wu, R. Ma, Z. Ai, X. Huan, S. Wang, W. Fan, Q. Zhang, In situ interfacial evaluation of aramid/epoxy composites by interfacial stress transfer characteristics, *Rev. Sci. Instrum.* 95 (7) (2024) 075110, <https://doi.org/10.1063/5.0211875>.
- [21] S.R. Wu, G.S. Sheu, S.S. Shyu, Kevlar fibre–epoxy adhesion and its effect on composite mechanical and fracture properties by plasma and chemical treatment, *J. Appl. Polym. Sci.* 62 (8) (1996) 1347–1360.
- [22] Y. Wang, X. Dong, Y. Wang, Z. Hu, Y. Chen, J. Yu, M. Zhu, Enhanced mechanical properties of aramid fibre/epoxy composites through reinforcing interfacial adhesion based on strong hydrogen bonding interactions, *Compos. Sci. Technol.* 262 (2025) 111057, <https://doi.org/10.1016/j.compscitech.2025.111057>.
- [23] S. Wang, C. Wang, Y. Chen, L. Liu, Z. Zhong, Y. Huang, M. Wang, Enhanced interfacial adhesion of aramid fibre composites by constructing a spider silk-inspired nanofibre network at interphase, *Compos. Sci. Technol.* 270 (2025) 111307, <https://doi.org/10.1016/j.compscitech.2025.111307>.
- [24] C. Lu, J. Wang, J. Wang, Enhancing interfacial adhesion: deprotonation and mussel-inspired coating of aramid fibres, *ACS Appl. Polym. Mater.* 6 (14) (2024) 109914, <https://doi.org/10.1021/acscpm.4c00723>.
- [25] J.-S. Lin, Effect of surface modification by bromination and metalation on Kevlar fibre–epoxy adhesion, *Eur. Polym. J.* 38 (1) (2002) 79–86, [https://doi.org/10.1016/S0014-3057\(01\)00176-8](https://doi.org/10.1016/S0014-3057(01)00176-8).
- [26] W. Guo, S. Lu, Z. Zhao, C. Sun, Y. Zhang, R. Qu, Y. Wang, Construction of aramid nanofibre-based interphase by in situ controlled polymerization and mechanism for interfacial reinforcement of aramid fibre composites, *ACS Appl. Mater. Interfaces* 18 (1) (2026) 2951–2966, <https://doi.org/10.1021/acscami.5c17351>.
- [27] G.J. Ehlert, H.A. Sodano, Zinc oxide nanowire interphase for enhanced interfacial strength in lightweight polymer fibre composites, *ACS Appl. Mater. Interfaces* 1 (8) (2009) 1827–1833, <https://doi.org/10.1021/am900376t>.
- [28] X. Kong, C. Hou, Y. Chen, Y. Du, G. Zhang, Y. Xing, B. Du, Simultaneously enhanced dielectric property and interfacial shear strength of aramid fibre reinforced epoxy composite via PDA-based coating with graphene oxide, *SSRN* (2024), <https://doi.org/10.2139/ssrn.4992953>.
- [29] G.S. Sheu, S.S. Shyu, Surface properties and interfacial adhesion studies of aramid fibres modified by gas plasmas, *Compos. Sci. Technol.* 52 (4) (1994) 489–497, [https://doi.org/10.1016/0266-3538\(94\)90031-0](https://doi.org/10.1016/0266-3538(94)90031-0).
- [30] W. Jing, F. Zhang, H. Chen, Comparative tribological performance and erosion resistance of epoxy resin composite coatings reinforced with aramid fibre and carbon fibre, *Colloids Surf. A Physicochem. Eng. Asp.* 648 (2022) 129354, <https://doi.org/10.1016/j.colsurfa.2022.129354>.
- [31] H. Dong, J. Wu, G. Wang, Z. Chen, G. Zhang, The ultrasound based interfacial treatment of aramid fibre/epoxy composites, *J. Appl. Polym. Sci.* 113 (3) (2009) 1816–1821, <https://doi.org/10.1002/app.30055>.

- [32] G. Li, C. Zhang, Y. Wang, P. Li, Y. Yu, X. Jia, H. Liu, X. Yang, Z. Xue, S. Ryu, Interface correlation and toughness matching of phosphoric acid functionalised Kevlar fibre and epoxy matrix for filament winding composites, *Compos. Sci. Technol.* 68 (16) (2008) 3208–3214, <https://doi.org/10.1016/j.compscitech.2008.08.006>.
- [33] M. Koyunbakan, Y. Uslugil, M. Ekrem, Ü. Eser, Investigation of the mechanical properties of aramid fibre-reinforced hybrid nanocomposites with BNNP-enhanced epoxy matrix, *Compos. Interfaces* 32 (12) (2025) 1711–1727, <https://doi.org/10.1080/09276440.2025.2496030>.
- [34] G.J. Ehler, Y. Lin, H.A. Sodano, Carboxyl functionalization of carbon fibres through a grafting reaction that preserves fibre tensile strength, *Carbon* 49 (13) (2011) 4246–4255, <https://doi.org/10.1016/j.carbon.2011.05.057>.
- [35] M. Shirazi, M.B. de Rooij, A.G. Talma, K.W.M. Noordermeer, Adhesion of RFL coated aramid fibres to elastomers: the role of elastomer latex compatibility, *J. Adhes. Sci. Technol.* 27 (17) (2013) 1886–1901, <https://doi.org/10.1080/01694243.2013.764145>.
- [36] J. Nasser, J. Lin, H. Sodano, High strength fibre reinforced composites with surface fibrilized aramid fibres, *J. Appl. Phys.* 124 (4) (2018) 045305, <https://doi.org/10.1063/1.5026987>.
- [37] L. Liu, Y.D. Huang, Z.Q. Zhang, Z.X. Jiang, L.N. Wu, Ultrasonic treatment of aramid fibre surface and its effect on the interface of aramid/epoxy composites, *Appl. Surf. Sci.* 254 (8) (2008) 2594–2599, <https://doi.org/10.1016/j.apsusc.2007.09.091>.
- [38] J.R. Brown, P.J.C. Chappell, Z. Mathys, Plasma surface modification of advanced organic fibres, *J. Mater. Sci.* 27 (12) (1992) 3167–3172, <https://doi.org/10.1007/BF02402964>.
- [39] H. Ulus, H.B. Kaybal, H.S. Sas, A. Avci, Flush-patch aluminum–FRP repair joints for construction and retrofitting: effects of laminate type, hybrid bonding and PAN nanofibre interleaves under pre-damage and hygrothermal ageing, *Int. J. Adhesion Adhes.* 148 (2026) 104300, <https://doi.org/10.1016/j.ijadhadh.2026.104300>.
- [40] H. Lee, S.M. Dellatore, W.M. Miller, P.B. Messersmith, Mussel inspired surface chemistry for multifunctional coatings, *Science* 318 (5849) (2007) 426–430, <https://doi.org/10.1126/science.1147241>.
- [41] T.S. Sileika, D.G. Barrett, R. Zhang, K.H. Aaron Lau, P.B. Messersmith, Colorless multifunctional coatings inspired by polyphenols found in tea, chocolate, and wine, *Angew. Chem. Int. Ed.* 52 (45) (2013) 10766–10770, <https://doi.org/10.1002/anie.201304922>.
- [42] J. Wu, L. Zhang, Y. Wang, Y. Long, H. Gao, X. Zhang, N. Zhao, Y. Cai, J. Xu, Mussel inspired chemistry for robust and surface modifiable multilayer films, *Langmuir* 27 (21) (2011) 13684–13694, <https://doi.org/10.1021/la2027237>.
- [43] S.M. Hosseini, M. Razzaghi Kashani, Vulcanisation kinetics of nano silica filled styrene butadiene rubber, *Polymer* 55 (30) (2014) 6426–6434, <https://doi.org/10.1016/j.polymer.2014.09.073>.
- [44] K.I. Garfias González, I. Odnevall Wallinder, K. Odelius, M. Hakkarainen, Surface modification of aramid fibre meshes – the key to chemically recyclable epoxy composites, *RSC Sustain.* 1 (8) (2023) 1967–1981, <https://doi.org/10.1039/D3SU00258F>.
- [45] H. Yuan, Z. Wang, D. Lan, et al., Aramid nanofibres at ultralow loadings: driving significant multifunctionality in epoxy composite dielectrics, *Adv. Compos. Hybrid Mater.* 8 (2025) 161, <https://doi.org/10.1007/s42114-025-01222-3>.