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Improving the interfacial adhesion between recycled carbon fibres and polyphenylene sulphide by bio-inspired dopamine for advanced composites manufacturing

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ABSTRACT

Polyphenylene sulphide composites with recycled carbon fibres are of high interest due to their promising mechanical properties. Poor interface adhesion between recycled carbon fibre and thermoplastic matrix, however, remains a major problem in composite manufacturing. Herein, a novel one-step method using bio-inspired copolymerisation of dopamine to graft silica nanoparticles onto the carbon fibre surface is reported. Comprehensive investigations were conducted to fabricate the treated carbon fibre and characterise the modified samples by analysing the surface morphology and functional groups. The experimental results reveal that the network of bio-inspired adhesive polydopamine and nanoparticles adhering to the fibres can improve interlaminar shear strength by 28.4% in the resulting composites. In addition, dynamical mechanical analysis results prove that the interfacial bonding at the fibre/matrix interface provides good resistance to thermal cycling. This novel approach introduces the functional groups on the recycled carbon fibre surface, leading to an improvement of surface energy and wettability between the carbon fibres and polyphenylene sulphide matrix and constitutes a robust and green approach to the manufacturing of advanced composites.

1. Introduction

Fibre-reinforced polymers (FRPs) have been widely used in aircraft structures due to their enhanced mechanical strength and light-weight for reduced fuel consumption (Stoeffler et al., 2013; Van de Werken et al., 2020). It was reported that the use of carbon fibres (CF) has increased from 48,500 tons in 2013 to 140,000 tons in 2020, expected to grow several times in size in the next decade (Karuppannan Gopalraj and Kärki, 2020). One of the most significant growth sectors is in the aerospace industry. Currently, more than 6000 aircraft will end their lives by 2030, thus there will be a big challenge to deal with the end-of-life composite materials because of the detrimental environmental impact and landfill area shortage (Zhang et al., 2020). Furthermore, a large amount of waste carbon fibres from out-of-life rolls of prepreg and off-cut the manufacturing process has already attracted public concern (Pimenta and Pinho, 2011; Yuan et al., 2017). Therefore, there is an urgent requirement to manufacture sustainable next-generation composites that not only mitigate the environmental impact but also maximise the economic value of recycled carbon fibre (rCF).

Technological breakthroughs in pyrolysis or fluidized bed process for recycling carbon fibre from thermoset composites, mainly epoxy resin, have recently made this product commercially available (Caltagirone et al., 2021; Hadigheh et al., 2021; Tian et al., 2022). While virgin carbon fibres offer higher mechanical strength, rCF is more cost-effective and environmentally beneficial. Challenges in using rCF to substitute virgin CF include weaker interfacial adhesion, processing challenges and potential unsuitability for high-performance applications. The integration of rCF and cost-effective polyphenylene sulphide (PPS) aims to address these issues, with a particular focus on enhancing interfacial adhesion, ensuring rCF/PPS composites meet the requirement of secondary structural components for the aerospace industry and extending its application to the automotive sector (Chen et al., 2022).

It is well known that the interface between carbon fibre and matrix is vital to the mechanical properties of composites because the bonding area is usually the weakest part of the whole structure (Wang et al., 2021; Wu et al., 2021a; Yu et al., 2014). However, non-polar carbon fibres lacking functional groups often lead to poor interfacial adhesion with thermoplastic matrices, which could result in severe fracture risks at the interface. The primary challenge includes the reduced mechanical properties, delamination, reduced fatigue resistance and compromised

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thermal resistance. Therefore, the weak interface properties limit the industrial application of carbon fibre/polyphenylene sulphide (CF/PPS) composites. Traditional surface modification methods include chemical modifications, physical treatments, and their combinations. Chemical modifications effectively enhance interfacial adhesion by introducing chemical groups onto the non-polar carbon fibre surface. Techniques such as grafting coupling agents (Zhu et al., 2021) and adding compatibilisers (Zhang et al., 2014) have been widely documented, increasing compatibility between carbon fibre and polymer and improving mechanical properties. Nonetheless, traditional chemical modifications often involve oxidation processes, organic solvents, and harsh reaction environments, potentially weakening the original carbon fibre and posing challenges for scaling up.

Plasma treatment, a key physical method, activates the carbon fibre surface efficiently. Low-pressure plasma techniques are promising for introducing functional groups such as ether, carboxylic, and carbonyl onto the carbon fibre surface, thereby enhancing interfacial adhesion (Tiwari et al., 2011). Despite their versatility and effective plasma-surface interaction through processing condition optimisation, these techniques have limitations, including inability to modify the carbon fibre bundle's inner surface, potential functional group degradation, and the requirement for complex vacuum equipment and high energy (Corujeira-Gallo and Dong, 2017).

Some researchers have utilised multiple chemical modification treatments or combined that with physical treatments in modifying the surface of the non-polar carbon fibre. Existing solutions generally follow a two-step approach: the first step is to roughen the smooth surface of carbon fibres, mainly including oxidation and plasma treatment, and the next step is to introduce a new layer between the fibre and matrix by adding compatibilizer and grafting coupling agents (Dong et al., 2020; Eyckens et al., 2021b; Hu et al., 2020; Wang et al., 2021). Previous research has explored various modification methods to enhance the interfacial strength. Despite the effectiveness, these conventional methods often rely on toxic chemicals, energy-intensive processed, or harsh operating conditions.

Increasingly, attention has shifted towards green solutions including advanced nanomaterials (Liu et al., 2018; Song et al., 2019; Yamamoto et al., 2019; Yang et al., 2007; Yao et al., 2018) and building green networks through bioinspired structures (Gao et al., 2019a; Liu et al., 2014a; Wu et al., 2023, 2022, 2021a, 2019; M. M. Zhang et al., 2020). The mussel's adhesion capability to various substrates, attributed to dihydroxyphenylalanine (DOPA), has inspired the use of polydopamine (PDA) for strong adherence to diverse surfaces, from inorganic to organic (Lee et al., 2007b). PDA's green synthesis in aqueous solutions and self-polymerisation process facilitate easy deposition on various surfaces (Feinberg and Hanks, 2022).

Recent research has focused on enhancing interfacial adhesion between commercial carbon fibre and thermosetting resins using PDA-assisted carbon fibre modifications. Gao et al. (2019b) reported a one-step copolymerisation of dopamine and poly(amidoamine) on carbon surfaces, markedly improving interfacial properties between carbon fibre and epoxy matrix. Wu et al. (Wu et al., 2019, 2021b, 2022, 2023) further explored this bioinspired strategy to deposit various layers (silane coupling agent, polyether amine or graphite oxide) on carbon fibre, offering a sustainable approach to fabricating enhanced-performance composites. Similarly, polydopamine assists in depositing silica nanoparticles (Ma et al., 2021) and Ni(OH)$_2$ nanosheets (Jin et al., 2020). This strategy has also been applied to thermoplastic resins, with hybrid sizing of carbon nanotube and polydopamine significantly enhancing the creep resistance of CF/PEI composites (Sun et al., 2022). Recently, to expedite PDA production, Sun et al. (2023) investigated an ultrasound-assisted PDA polymerisation process on CF, also facilitating PDA molecule permeation into the fibre bundle and highlighting the crucial role of phenolic hydroxyl and amino groups for compatibility with the PA6 matrix.

While numerous studies have focused on using PDA to modify the surface of carbon fibres and to graft polymer or nanomaterials onto them (Jin et al., 2020; Ma et al., 2021; Sun et al., 2022; Wu et al., 2019, 2021a, 2022, 2023), existing literature has not yet bridged the gap between this novel, environmentally friendly approach, and its application to recycled carbon fibres and high-temperature thermoplastic resins like polyphenylene sulphide. In response, this work develops a more sustainable one-step surface modification strategy that employs PDA as an adhesive agent for recycled carbon fibres. This PDA layer forms a hybrid network with silica nanoparticles on the fibre surface, acting as a multi-functional interface. This interface not only enhances adhesion but also introduces new mechanical and chemical pathways for stress distribution, thereby improving the performance of rCF/PPS composites. Our work takes a significant step toward lowering the environmental footprint of composite manufacturing.

2. Materials and methods

2.1. Materials

Carbon fibre (woven fabric cloth, 3K) was purchased from Easy composites Ltd, Stoke on Trent, UK. Tetraethoxysilane (TEOS), Dopamine hydrochloride (DP) and Tris (hydroxymethyl)-aminomethane (Tris) were purchased from Merck Life Sciences Ltd, UK. Polyphenylene sulphide (PPS, Torelina A900) was manufactured by Toray Industries, Japan. Acetone used in the desizing process was obtained from Fisher Scientific Ltd, Leicestershire, UK. All chemicals were used as received unless stated otherwise.

2.2. Fabrication of CF-PDA/NPs

Colloidal silica nanoparticles (NPs) were synthesised using tetraethyl orthosilicate (TEOS) as the precursor following a specified route (Ibrahim et al., 2010). Initially, a mixture of ethanol and TEOS was prepared to form solution A, while solution B comprised a mixture of ethanol, deionised water, and ammonia solution. Subsequently, solution B was gradually added to solution A at a temperature of 60 °C. Ammonia served as a catalyst to expedite the hydrolysis and condensation reactions. The concentration of ammonia dictated the pH of the solution, which in turn significantly influenced the size and distribution of the silica nanoparticles. Upon introduction to water, TEOS promptly undergoes hydrolysis catalysed by ammonia, resulting in the formation of silicic acid (Si(OH)$_4$). These silicic acid molecules then undergo polycondensation to form silica nanoparticles. The resultant colloidal silica nanoparticles purified through centrifugation, followed by concentration using a rotary evaporator. Transmission electron microscopy (TEM) image for silica nanoparticles shows that the average diameter of a single particle is around 50 nm (Fig. 1(c)), which is dispersed uniformly in the buffer solution.

Untreated (as-received) carbon fibre was desized by acetone first to remove any contaminants or sizing agents (Fig. 1(a)), and then the concentrated silica nanoparticles were dispersed in the buffer solution (0.1% Tris solution, pH = 8.5), which is suitable for the self-polymerisation reaction of dopamine. The carbon fibre and dopamine hydrochloride were added to the buffer solution simultaneously at room temperature with an ultrasound instrument to produce the treated carbon fibre CF-PDA/NPs, as illustrated in Fig. 1(b). After immersing the fabric cloth in the modification solution, it was removed and rinsed three times with ultra-pure water and then dried at 60 °C in the oven for 48 h to obtain the treated CF. As seen in Fig. 1(d), nanoparticles were coated through the self-polymerisation of dopamine reacting in the buffer solution, followed by depositing themselves on the carbon fibre surface, to ensure the PDA layer and silica nanoparticles are securely attached.

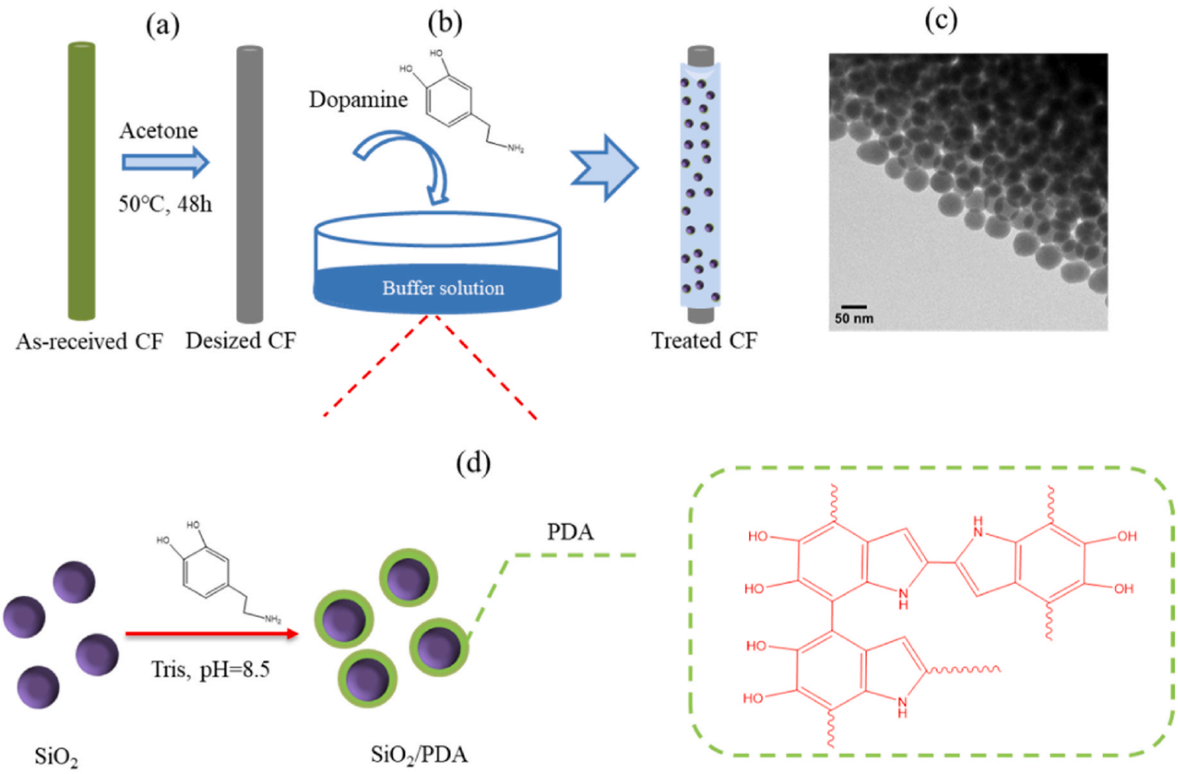


Fig. 1. Fabrication scheme of the treated carbon fibre: (a) desizing procedure; (b) dopamine/silica nanoparticles treatment; (c) silica nanoparticles; (d) reaction scheme in buffer solution.

2.3. *Characterisation of carbon fibres*

Scanning electron microscope (SEM) instrument (JEOL, JSM-IT100) was applied in observing the surface morphology of the desized carbon fibre and treated carbon fibre. Samples were initially dispersed in ethanol and subsequently evaporated onto stubs at room temperature. Following this, the sample underwent sputter-coating with a conductive material, specifically gold, to enhance conductivity. SEM images were captured at magnifications of × 4000 to meticulously assess the distribution, density, and adherence of silica nanoparticles on the carbon fibre surface.

Transmission Electron Microscopy (TEM), employing a JEOL JEM1400 Plus instrument, facilitated detailed observations of the polydopamine and nanoparticle coatings on individual fibres. Carbon fibres were dispersed in ethanol, evaporated onto a mesh for TEM examination. This allowed for an in-depth analysis of the coatings' cross-sections and the interface between the desized carbon fibre, polydopamine layer, and silica nanoparticles.

Raman spectroscopy was employed to assess near-surface structural variations across different fibres. A Renishaw inVia Raman microscope was applied to generate the Raman spectra of the different carbon fibre surfaces. The spectra were obtained using excitation laser lines of 514 nm, with a spectral resolution of 1 cm^{-1}.

X-ray photoelectron spectroscopy (XPS, Scienta 300) spectra were performed determine the elemental composition and chemical states of atoms within the carbon fibre surface. The X-ray source is a SPECS monochromated Al Ka source (photon energy 1486.6 eV) operating at approx. 12 KV and 200 W power. For all spectra, the instrument maintained a pass energy set to 150 eV. Survey scans were collected with a dwell time of 133 msec, a step size of 200 meV, and 2 scans aggregated for enhanced accuracy.

Fourier Transform Infrared (FTIR, Shimadzu, IRTracer-100) spectroscopy was used to characterise the presence of the functional groups of dopamine hydrochloride, polydopamine, silica nanoparticles and polydopamine/silica nanoparticles. Each sample was scanned 60 times in the settings for wavenumbers between 4000 cm^{-1} and 500 cm^{-1}.

2.4. *Preparation of the CF/PPS composite laminates*

Carbon fibre woven fabrics were hot pressed into the PPS matrix to form CF/PPS laminates using a PEI Lab 450 press. The PPS pellets were milled into powders (70 μm in average) first and then pressed at elevated temperatures in a mould to fabricate PPS films. Then the PPS film was compounded with carbon fibre using the film stacking method to produce CF/PPS sheets. Lastly, 9 plies of CF/PPS composite sheets were placed in the mould again to complete the consolidation process by hot press. The time-temperature-pressure cycle of the hot press is shown in Fig. S1 and was plotted with parameters referenced in (Grouve and Akkerman, 2010).

2.5. *Interlaminar shear strength of CF/PPS composite laminates*

To investigate the interfacial adhesion between woven carbon fibres and PPS matrix, interlaminar shear strength (ILSS) was measured by the short beam test according to the ASTM D2344 standard. The short beam specimens were cut from the hot-pressed CF/PPS composite laminates with the dimensions of 24 mm × 8 mm × 4 mm. The tests were carried out on an Instron 3369 universal testing machine, where the span length was 16 mm and the crosshead rate was 1.0 mm/min. Three tests were repeated for each type of composite laminate.

2.6. *Dynamic mechanical analysis (DMA) of CF/PPS composite laminates*

The thermal-mechanical behaviours of resulting CF/PPS laminates were studied on a TA instrument Discovery DMA 850 using the dual cantilever mode. Specimens with dimensions of 55 mm × 13 mm × 3 mm were tested at a frequency of 1 Hz, and the oscillation amplitude was set to 30 μm recommended by relevant standard (Menard and Menard, 2020). The heating rate was 2 °C/min from 35 °C to 250 °C in a nitrogen environment to avoid any degradation (Kishore et al., 2020). The DMA specimens, containing 6 plies of CF/PPS sheets, were manufactured by a hot press.

3. Results and discussion

3.1. Surface morphology of carbon fibres

To observe the copolymerisation process of dopamine and nanoparticles in the modification process, SEM was used to characterise the surface morphology of desized carbon fibre and treated carbon fibre. Samples were prepared at different coating times, *i.e.*, 2 h, 4 h, 14 h, 16 h and 18 h, respectively. In this study, the nanoparticles concentration is set to 2% and the dopamine concentration is 0.1%. As expected, Fig. 2 shows that as the treating time increased, the surface topography of functionalised carbon fibres was distinctly different from the smooth and neat surface of desized carbon fibres. Also, the surface roughness increased over time. A uniform layer with many nanoparticles was formed on the carbon fibre surface after 16 h of treatment. It is interesting to note that the surface morphology of the carbon fibre surface no longer changed when the modification time reached 18 h. Results illustrate that the self-polymerisation process of dopamine continuously reacts as the coating time increases and remains stable for more than 16 h under these conditions. Therefore, the optimal modification time is set as 16 h, which is applied in the following experiments.

In addition, the synergistic effect of the polydopamine (PDA) and nanoparticles (NPs) plays a key role in the interfacial properties of carbon fibre and PPS polymer matrix. For comparisons, the specimens treated by 0.1%PDA solution (CF-PDA), 2%NPs solution (CF-NPs), 0.1% PDA-

1%NPs solution (CF-PDA/NPs1), 0.1%PDA-2%NPs solution (CF-PDA/NPs2) and 0.1%PDA-4%NPs solution (CF-PDA/NPs4), respectively, were also prepared to evaluate the difference in surface morphologies by TEM. Fig. 3 displays the surface morphologies of carbon

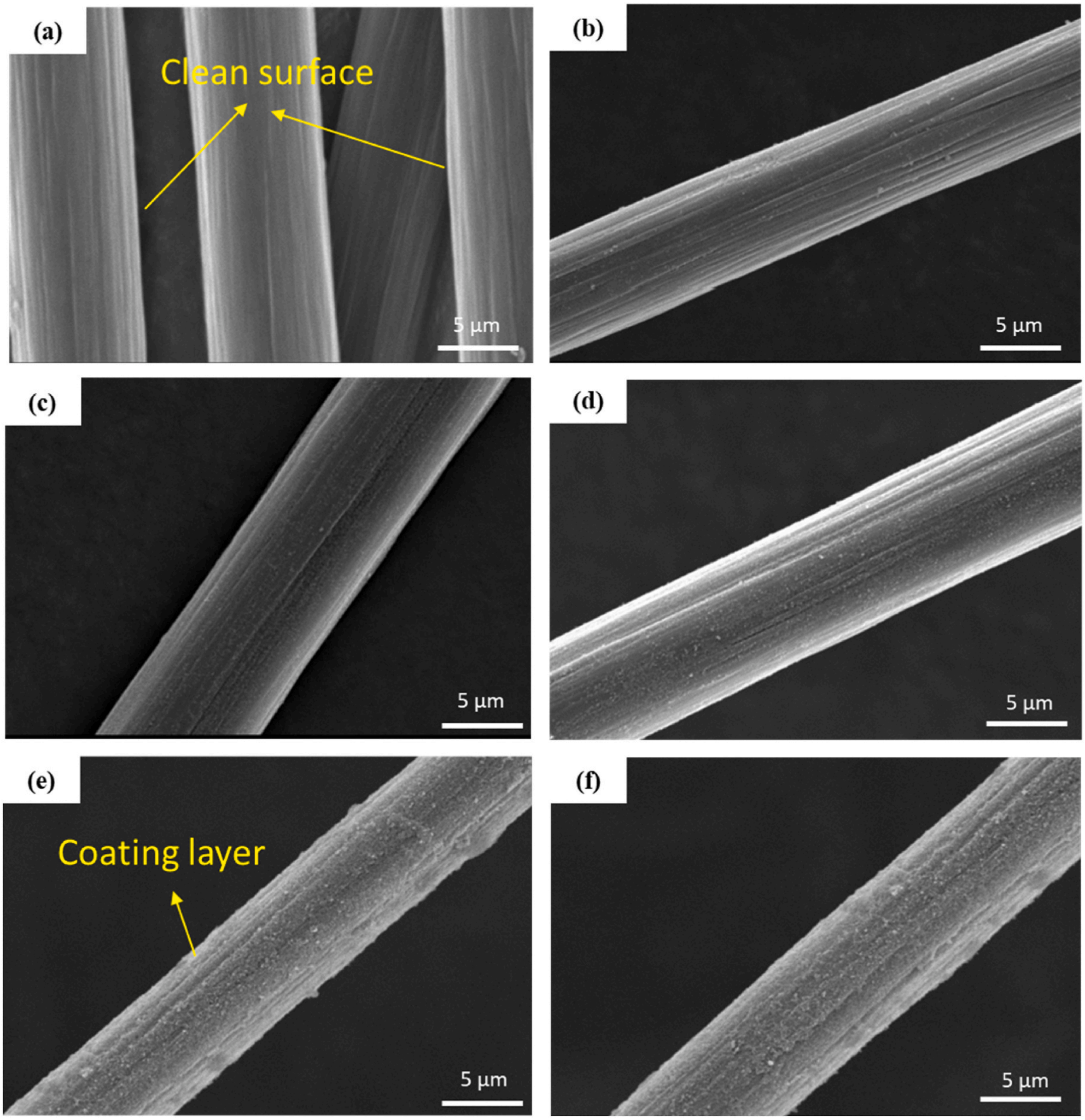


Fig. 2. Surface morphology of carbon fibre at different treatment times: (a) 0 h (b) 2 h; (c) 4 h; (d) 14 h; (e) 16 h; (f) 18 h. The nanoparticles concentration is set to 2% and the dopamine concentration is set to 0.1%.

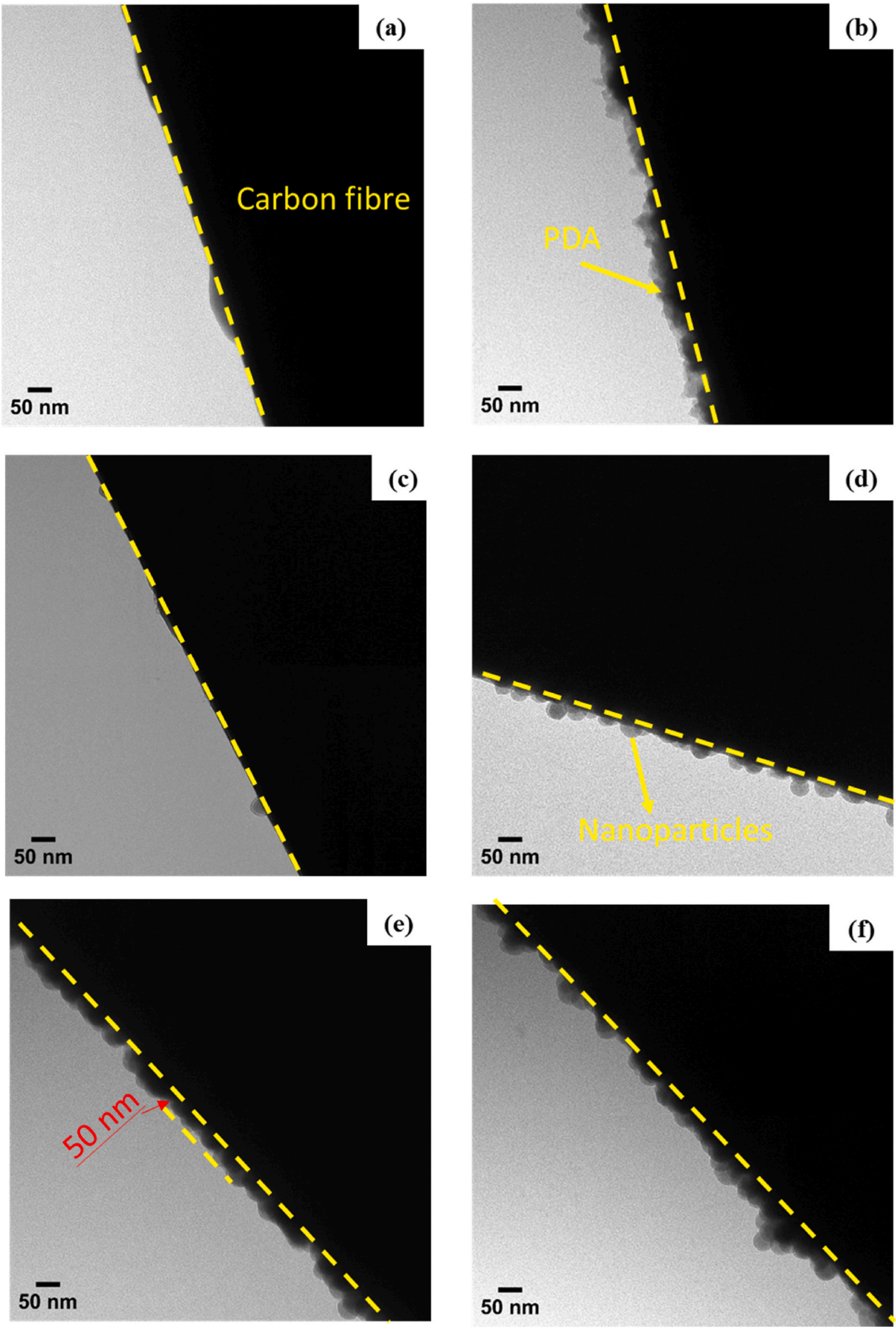


Fig. 3. Surface morphology of carbon fibre with different treated solutions (treatment time: 16 h): (a) desized carbon fibre (b) 0.1%PDA solution (CF-PDA); (c) 2% NPs solution (CF-NPs); (d) 0.1%PDA-1%NPs solution (CF-PDA/NPs1); (e) 0.1%PDA-2%NPs solution (CF-PDA/NPs2) and (f) 0.1%PDA-4%NPs solution (CF-PDA/NPs4).

fibres treated by these different solutions. Compared with the sole CF-PDA, a distinct layer of nanoparticles (around 50 nm) was observed on the carbon fibre surface in the CF-PDA/NPs2 as well as CF-PDA/NPs4. Furthermore, a more homogenous deposition layer of nanoparticles was obtained in CF-PDA/NPs1 than CF-NPs. They were likely deposited and grafted on the surface with the assistance of the PDA. Interestingly, as the concentration of nanoparticles continuously increases, however, the surface morphologies did not show significant differences between the 0.1%PDA-2%NPs and 0.1%PDA-4%NPs solution. Results indicate that a solution concentration of 0.1% PDA and 2% colloidal nano-SiO_2 solution is most suitable for treatment of carbon fibres due to its ability to create a uniform adhesive layer on the CF surface, offering an ideal balance between morphological enhancement and cost-effectiveness. Overall, in the modification process, the polydopamine acting as an efficient and robust platform could support the grafting of the nanoparticles. The mechanism of the possible copolymerisation process is explored in the later section.

3.2. Chemical analysis of modified carbon fibres

3.2.1. Raman spectroscopy

Raman Spectroscopy was used to evaluate the surface structural variations because it is sensitive to the order structure. Fig. 4 (a) shows the Raman spectra of desized CF, CF-PDA, CF-NPs, CF-PDA/NPs2 and CF-PDA/NPs4. There are two distinct peaks displayed in the spectroscopy D band (around 1370 cm^{-1}) and G band (around 1600 cm^{-1}), respectively. As it is well known, the D band is a result of the structure disorder or defects in graphite, while the G band is due to in-plane vibration of sp2 bonded carbon atoms corresponding to ordered graphite structure (crystallinity) (Wu et al., 2021a). Compared to the desized carbon fibre, the peak intensity of D and G band for carbon fibres treated by PDA and NPs solely show minor difference. However, the peak intensity of D and G band was obviously increased by the incorporation of both polydopamine and nanoparticles. CF-PDA/NPs2 exhibits the highest intensity of the D peak and G peak.

The intensity ratio of the D band and G band reveals the functionalisation degree and the extent of structural defects or disorders (Fang et al., 2009). A higher ID/IG ratio indicates more defects or disorder in the carbon material. For desized CF, the ID/IG ratio is 1.973, which is relatively high, suggesting a significant level of disorder, which is common in carbon fibre that have been desized to remove the sizing agents during the manufacturing process. For CF-PDA, this sample has been treated with polydopamine, presenting a highest ID/IG ratio (2.648), which indicates PDA treatments could introduce additional defects or disorder into the carbon structure, due to the functionalisation or roughening of the surface as a result of the PDA coating. For the CF-NPs, the ID/IG ratio of 2.511 is also higher than desized carbon fibre, suggesting a higher disorder on the carbon fibre surface, which could result from the non-unform deposition of a few nanoparticles aggregates on the surface of carbon fibre. Once PDA is introduced, the ID/IG ratios are lower at 1.508 and 1.425 for CF-PDA/NPs-2 and CF-PDA/NPs-4, respectively. This decrease in the ID/IG ratio compared to the CF-NPs sample could indicate that the combined treatment creates a stronger π-orbital hybridization with a more ordered structure than nanoparticle treatment alone, supporting the assertion that PDA could increase the intact graphitic domains (Wu et al., 2019). As expected, the result shows evidence that nanoparticles were successfully grafted onto the surface of the treated CF assisting with polydopamine to produce the CF-PDA/NPs. Further analysis of chemical groups will be combined with X-ray photoelectron spectroscopy (XPS) in the later section.

3.2.2. X-ray photoelectron spectroscopy

X-ray photoelectron spectroscopy (XPS) was applied to measure the changes in the binding energy of the ejected photoelectrons in order to elucidate the modifications in chemical compositions of the treated CF, compared to desized CF. Fig. 5 depicts the results of survey scans for desized CF and fabricated CF-PDA/NPs2. Three peaks assigned to C, N and O elements were found in the survey spectra of desized CF, while one extra Si element was observed in the treated CF sample (enlarged peak). To get more detailed information, the peak fitting results in the C 1s spectrum and N 1s spectrum of desized CF and fabricated CF-PDA/NPs2 were also displayed in Fig. 6. These core-level spectra were fitted with several peak components separately based on the binding energies.

In Fig. 6(a), there are four peaks assigned to C–C bonding (284.6 eV), C–N bonding (285.7 eV), C–O bonding (286.6 eV) and O═C–O bonding (288.0 eV) respectively in C 1s spectra of desized CF (Ech-Chamikh et al., 2006; Yang et al., 2009). Compared with the desized sample, there emerged a new peak attributed to C═O bonding (287.6 eV) (Gao et al., 2019b; Yang et al., 2009) in the spectra of the treated sample, as shown in Fig. 6(b). Additionally, the peak area fraction of the nitrous carbons C–N and oxygenated carbons C–O shows a great increase from 27.1% of desized CF to 50.2% of fabricated CF-PDA/NPs2. Further, the detailed information on differences of N 1s core-level spectra between the desized CF and treated CF. As presented in Fig. 6(c), two fitted peaks represent the N–H bonding and C–N bonding with the binding energy of 397.8 eV and 400.1 eV respectively (Dong et al., 2020; Ech-Chamikh et al., 2006). As for CF-PDA/NPs2, the spectra are decomposed into three peaks with a new peak position with the binding energy of 402.5 eV arising from the N–SiO_2 structure (Shallenberger et al., 1999). In addition, the content N–H increased from 7.6% of desized CF to 12.7% of fabricated CF-PDA/NPs2. By the introduction of the PDA/NPs network, these polar nitrogen-containing functional groups, especially for –NH_2, play an important role in activating the carbon fibre surface (Gao et al., 2019b), which could further react with the matrix to increase the interfacial strength between carbon fibre and PPS matrix in the resulting composites. The reaction mechanism is analysed in Section 3.4.

3.3. Mechanical performance of the resulting CF/PPS composites

3.3.1. Interlaminar shear strength (ILSS)

The short-beam test is an effective way to evaluate the interfacial properties of fibre-reinforced composites. In Fig. 7, the ILSS values of composites with untreated CF and desized CF are quite close (17.50 MPa and 18.43 MPa, respectively), which indicates that the interfacial bonding between the carbon fibre and PPS matrix is almost the same after the desizing of the carbon fabrics. The untreated carbon fibre was desized first and then treated with a solution containing 0.1% PDA and 2% colloidal nano-SiO_2 to obtain the treated CF. After the establishment of the network of CF-PDA/NPs on the carbon fibre surface, the ILSS value reaches 23.66 MPa with a 28.4% increase compared to that of desized CF composites. By comparison with the previous studies which shows increases of between 15% and 40% (Hu et al., 2020; Liu et al., 2018; Song et al., 2019; Yang et al., 2007; Yao et al., 2018), this one-step modification strategy has advantages in low cost, low energy consumption and is also free of toxic chemicals. Overall, the figure effectively demonstrates that the treatment of carbon fibres with polydopamine and nanoparticles leads to a substantial improvement in their mechanical performance, as evidenced by the increased ILSS values. This treated CF exhibits not only better performance compared to the untreated and desized counterparts but also shows significant potential for use in advanced composite materials where higher mechanical strength is required.

3.3.2. Dynamic mechanical analysis

To enhance our understanding of the thermo-mechanical behaviour of the treated carbon fibre (CF) reinforced polyphenylene sulphide (PPS) composites, dynamic mechanical analysis (DMA) was conducted over a temperature range of −60 °C–250 °C. Fig. 8 presents the storage modulus (G') alongside the damping factor (tan δ) for both untreated and treated CF composites. Notably, the treated composites exhibit

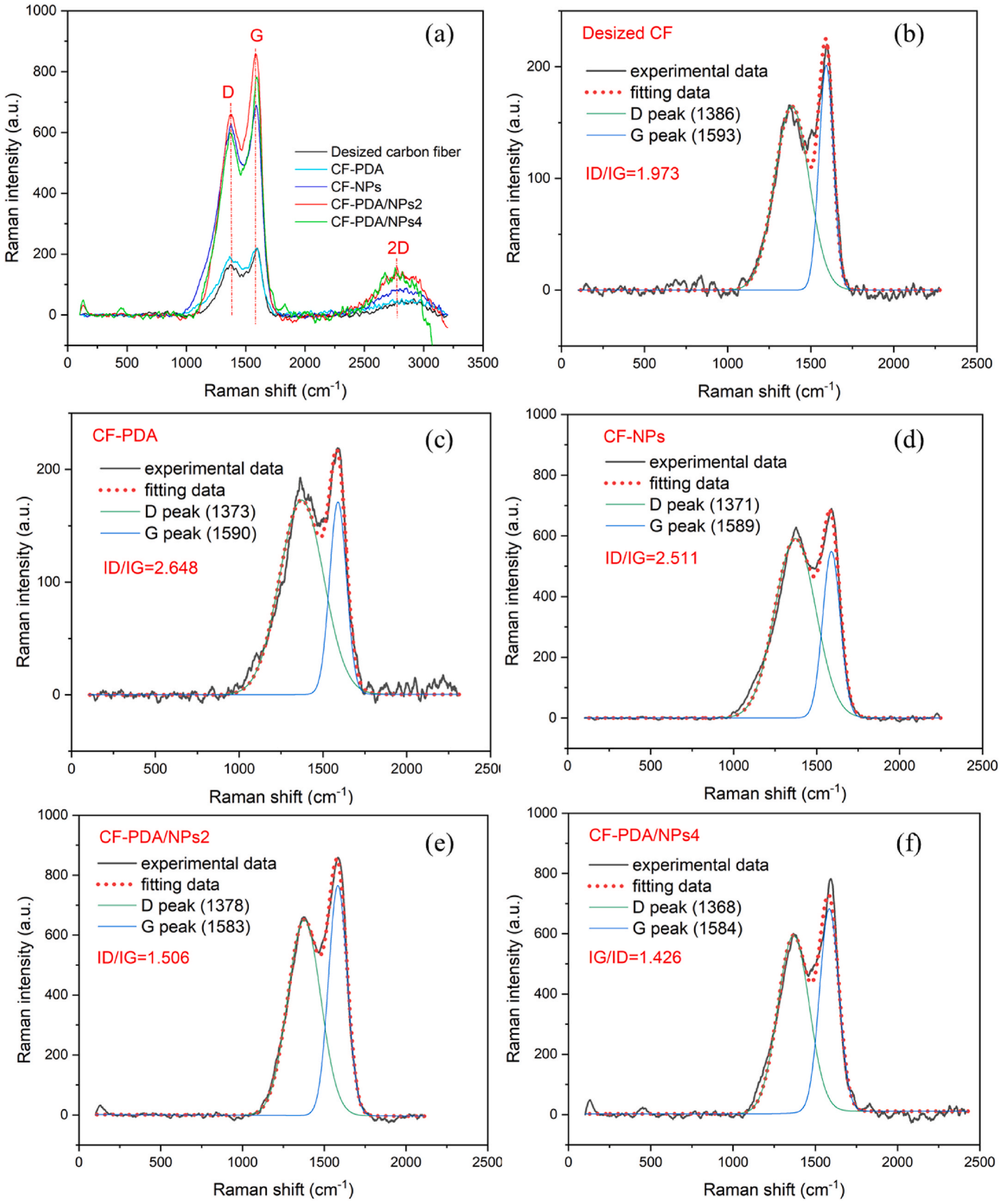
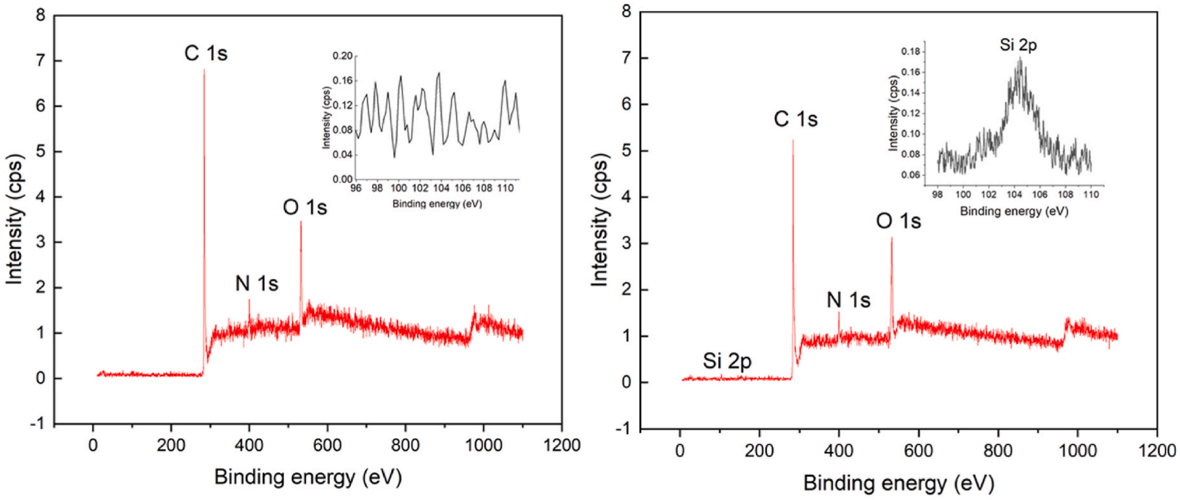


Fig. 4. Raman spectroscopy (a) and peak fitting results of carbon fibres: (b) desized carbon fibre (c) 0.1%PDA solution (CF-PDA); (d) 2%NPs solution (CF-NPs); (e) 0.1%PDA-2%NPs solution (CF-PDA/NPs2) and (f) 0.1%PDA-4%NPs solution (CF-PDA/NPs4).



(a) (b)

Fig. 5. Survey scans of (a) desized CF and (b) fabricated CF-PDA/NPs2.

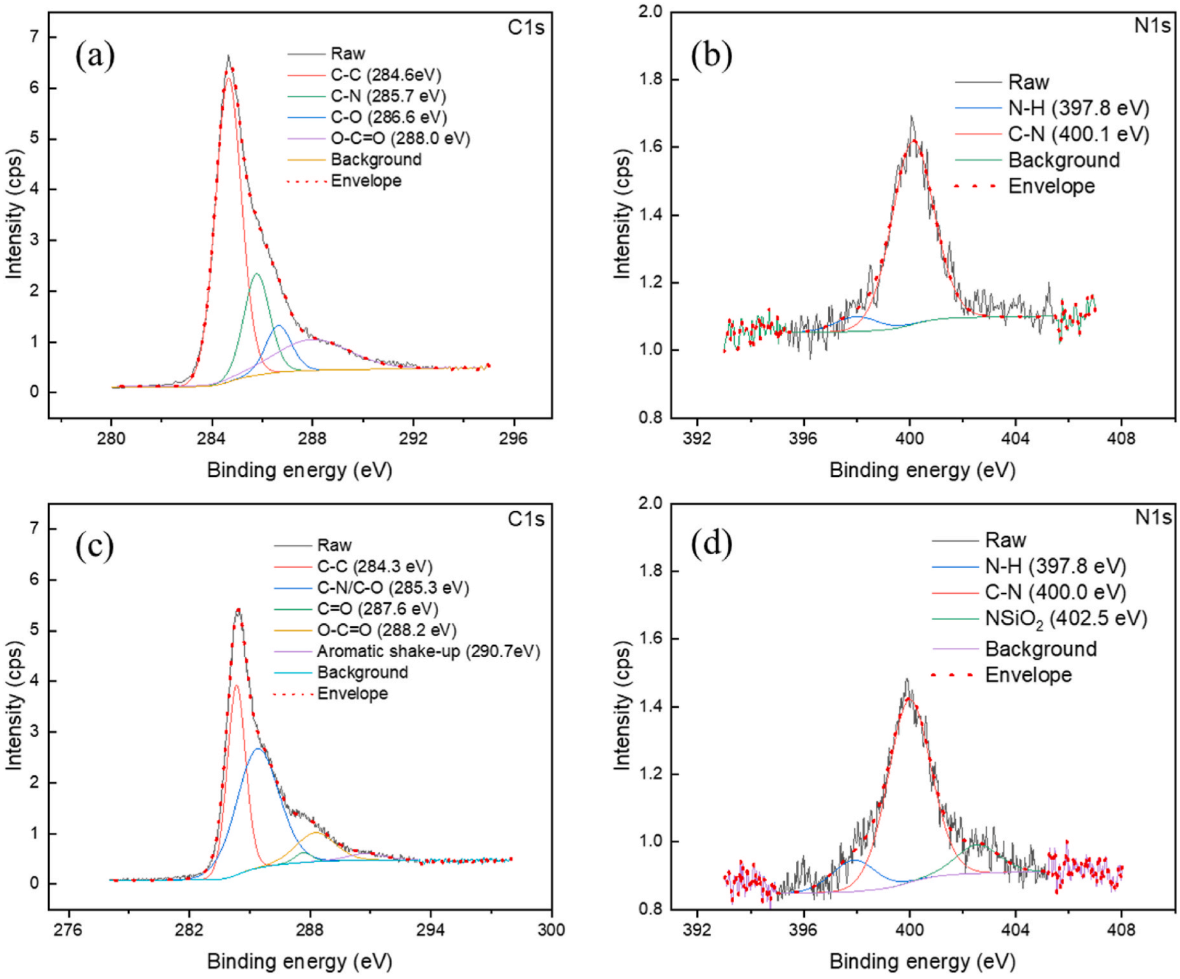


Fig. 6. C1s and N1s spectra of desized CF (a & b) and fabricated CF-PDA/NPs2 (c & d).

consistently superior G' values across the entire temperature spectrum, with an improvement of 15% in G' at ambient temperatures post-treatment, rising from 4687 MPa for untreated CF to 5380 MPa for treated CF in the initial cycle.

Moreover, the damping factor (tan δ) reveals an obvious difference between the treated and untreated samples. This factor is a recognised indicator of the energy dissipation mechanisms within a material (SudarshanRao, 2021). Reduced damping factor in the treated

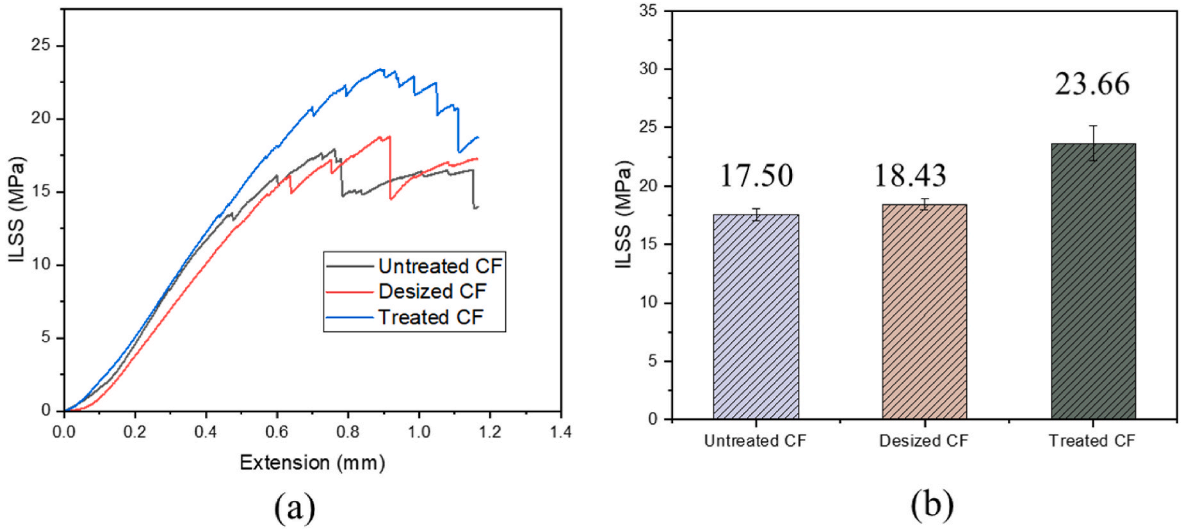


Fig. 7. Interlaminar shear strength (ILSS) of the resulting composites manufactured with untreated CF, desized CF and treated CF: (a) representative ILSS versus extension curves (b) ILSS results and deviations.

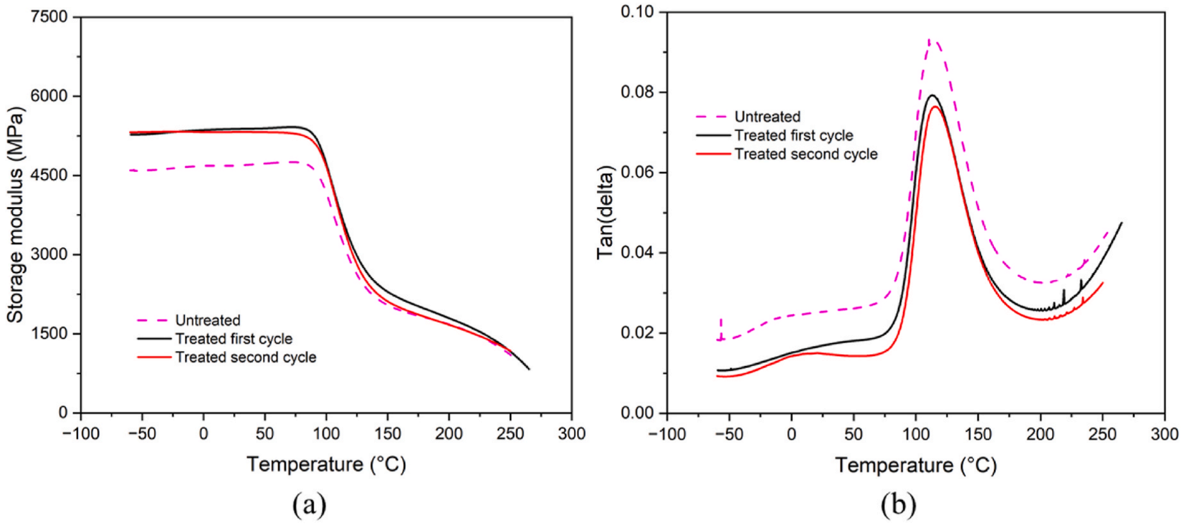


Fig. 8. DMA results of the resulting composites manufactured with untreated CF and treated CF: (a) storage modulus and (b) damping factor.

composites suggests that higher energy is used to deformed the treated composites, indicative of an improved fibre/matrix interface. The results demonstrate that the PDA/NPs network could play an important role in improving the viscoelastic behaviour of the resulting composites.

Furthermore, the robustness of the interfacial bonding at the matrix/fibre interface was put to the test through a second series of thermal heating-cooling cycles. The subsequent findings, also depicted in Fig. 8, confirm the exceptional thermal resistance of treated composites, particularly between −60 °C and 100 °C. Remarkably, even under thermal cycling for extreme conditions, the treated interface retains 99% of its initial performance at 100 °C, to be superior to the virgin interface. This result shows that the thermal cycles have negligible effect on the storage modulus of the resulting CF/PPS composites at serviced temperatures, which for potential applications in environments where materials are subjected to extreme and fluctuating temperatures.

In conclusion, the DMA results clearly reveal that the PDA/NPs treatment enhances fibre/matrix bonding, as evidenced by increased storage modulus (G'), leading to stiffer composites with superior load handling. Moreover, the composites exhibit remarkable resilience to thermal cycling, preserving most of their original storage modulus after repeated cycles. These findings are particularly relevant for applications in harsh thermal environments, where material performance and longevity are of utmost importance. These properties are crucial for high-performance applications requiring materials that can withstand harsh operating conditions during repeated heating and cooling cycles without compromising their structural integrity or performance.

3.4. Mechanism for the improvement of interfacial properties by the PDA/NPs network

Fig. 9 presents the FTIR spectra of dopamine hydrochloride, polydopamine, silica nanoparticles (NPs), and polydopamine-functionalised silica nanoparticles (PDA/NPs). The spectral analysis in Fig. 9(a) highlights the variances in functional groups between dopamine and its polymerised form, polydopamine. The polydopamine spectrum exhibits an obvious broad peak between 3000 and 3500 cm^{-1}, reflective of O–H and N–H stretching vibrations. Additionally, peaks around 2900 cm^{-1} correspond to the C–H stretching vibrations, a feature also present in dopamine hydrochloride spectra. Notably, new peaks at 1627 cm^{-1} and 1552 cm^{-1}, which appear from the 1616 cm^{-1} band, are attributable to

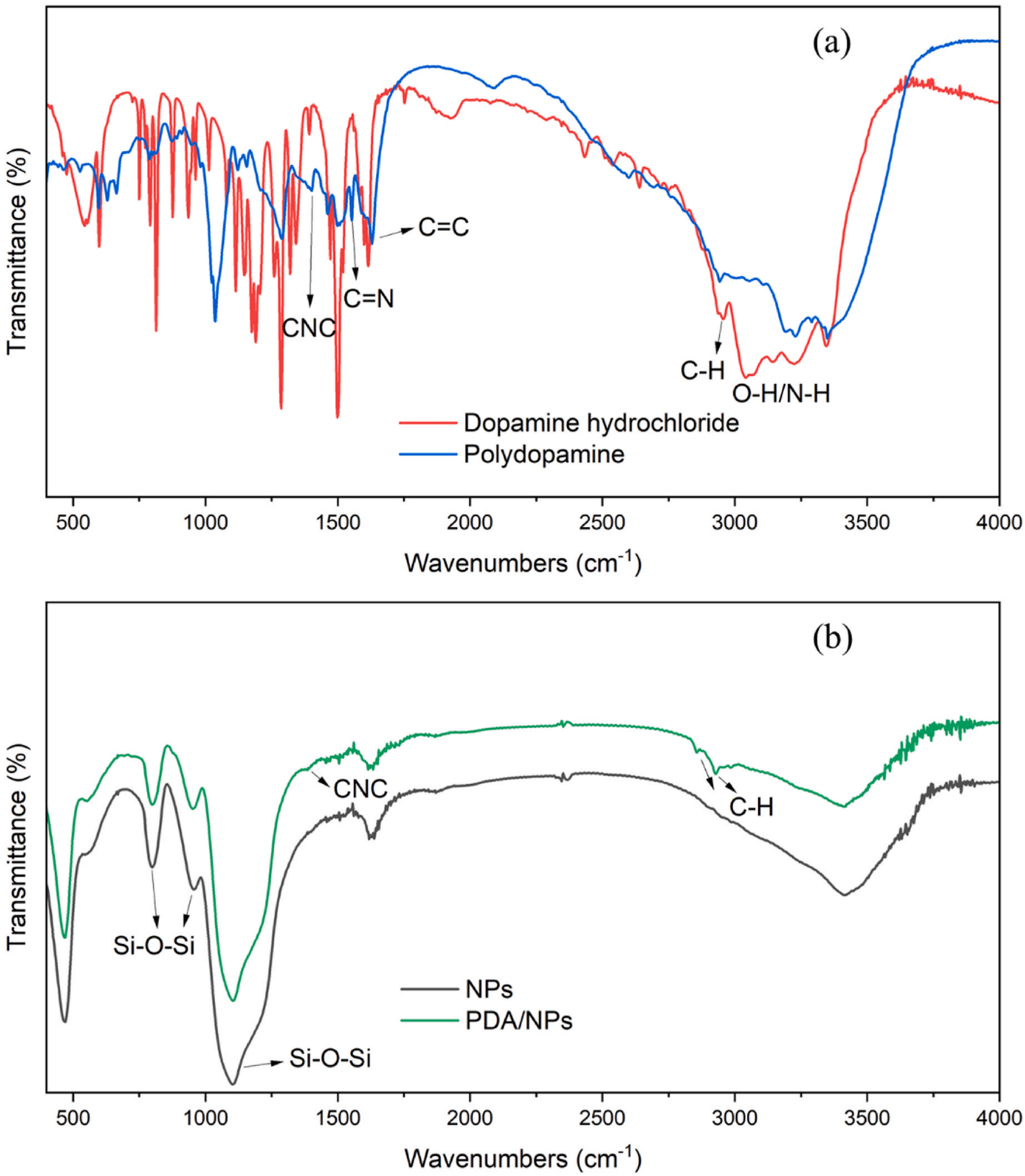


Fig. 9. FTIR spectrum of (a) dopamine hydrochloride and polydopamine (b) silica nanoparticles (NPs) and polydopamine functionalised silica nanoparticles (PDA/NPs).

the C=C and C=N stretching vibrations in aromatic rings. The emergence of another peak at 1400 cm^{-1} is identified with the indole ring CNC stretching modes, corroborating the existence of 5,6-dihydroxyindole units within the structure (Dreyer et al., 2012; Zangmeister et al., 2013)

Fig. 9(b) exhibits FTIR spectrum of silica nanoparticles (NPs), and polydopamine functionalised silica nanoparticles (PDA/NPs). For both structures, the peaks observed at 798 cm^{-1} and 960 cm^{-1} are characteristic of the Si–O–Si bending vibrations, and the peak at approximately 1100 cm^{-1} is indicative of Si–O–Si stretching vibrations. These peaks are typical for silica and confirm the presence of Si–O bonds (Panwar et al., 2015). After functionalisation with PDA, additional peaks at 2924 cm^{-1} and 2856 cm^{-1} attributed to the C–H stretching vibrations from the PDA, and the appearance of the characteristic peak at 1400 cm^{-1} confirms that the PDA layer has been successfully grafted onto the silica nanoparticles.

Fig. 10 presents the fabrication process of PDA-assisted silica nanoparticles coated on the carbon fibre surface and the following reaction pathway in the resulting composites, to understand the possible reaction mechanism between the CF-PDA/NPs and PPS matrix. Based on previous studies, the typical polymerisation process of the PDA is presented for the in-situ polymerisation of its monomer, dopamine, which can be oxidized by oxygen and self-polymerised in the alkaline solution (Jiang et al., 2011; Liu et al., 2014b). This spontaneous polymerisation reaction is green and mild without any harsh reaction conditions. When dopamine hydrochloride is dissolved under alkaline conditions (pH > 7.5),

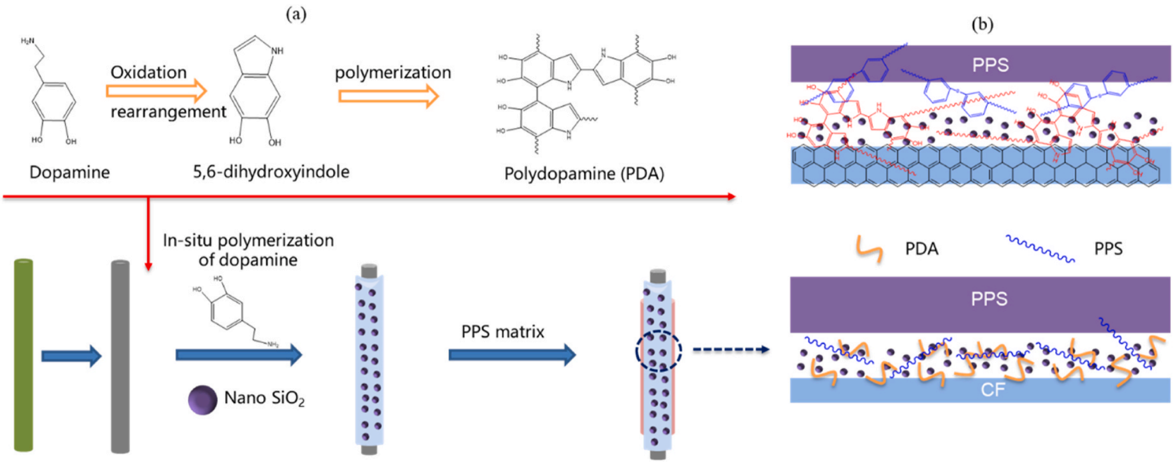


Fig. 10. (a) Functionalisation of PDA/NPs on the carbon fibre surface and (b) possible reaction mechanism between CF-PDA/NPs and PPS matrix in the resulting composites.

this chemical reaction immediately occurs in the buffer solution. The colourless solution will turn yellow-brown first and finally change to dark brown. The dopamine suffers from oxidation first to form dopamine-quinone, followed by intramolecular cyclisation through Michael Addition to produce leucodopaminechrome, which is further oxidized to 5,6-dihydroxyindole. This reaction product can be easily oxidized into 5,6-indolequinone, forming the cross-linked polymer (Liu et al., 2014a; Yang et al., 2014).

It has been reported that polydopamine has strong adhesion to almost all types of surfaces (Lee et al., 2007a). Overall, the mechanisms are generally divided into two types of bonding: covalent bonding and non-covalent bonding (Feinberg and Hanks, 2022). Typically, covalent bonding can be formed on substrates containing some specific groups, such as the amine group via Michael addition or Schiff base. In most cases, however, PDA tends to diffuse into the surface to form non-covalent bonding including chelating, hydrogen bonding, π-π stacking, and quinhydrone complex, especially for the interactions under ambient conditions (Bliznyuk et al., 2019). In this study, the polymerisation of dopamine on the carbon fibre surface introduces a variety of functional groups, notably amine and catechol groups. These functional groups serve multiple roles, including acting as binding sites for silica nanoparticles. The catechol group of PDA could potentially forms the hydrogen bonding with the hydroxyl groups in silica nanoparticles (Yang et al., 2014). The presence of these functional groups enhances the chemical affinity between the carbon fibres and silica nanoparticles. Polydopamine layer coated on the carbon fibres provides an active surface for further deposition of silica nanoparticles. As a result, the PDA-coated nanoparticles could be easily adhered to the activated surface of carbon fibres by the non-covalent bonds under ambient conditions.

Polydopamine forms a cohesive and adhesive layer on the surface of carbon fibres, consisting of an interconnected network of dopamine monomer units that have undergone oxidative polymerisation. The PDA layer acts as a foundation for nanoparticles attachment, through various types of chemical interactions, such as hydrogen bonding. Nanoparticles can be embedded into or layered onto the PDA layer. The resulting structure is a cross-linked network where the PDA binds the nanoparticles to the carbon fibre surface, enhancing the topography and increasing the functional group density. The PDA/NPs network could significantly enhance the adhesion between carbon fibres and the matrix material in composites. The presence of various functional groups like catechol and amine groups allows for strong interfacial bonding with PPS matrix molecules (Feng et al., 2022). Specifically, catechol can interact with ant polar sites on the PPS, while amine group can form hydrogen bonds with oxygen-containing groups. The aromatic rings present in both PDA and PPS can engage in π-π stacking interactions. Moreover, the integration of nanoparticles within the PDA layer increases the surface roughness of the carbon fibres, amplifying van der Waals forces with increased contact area and promoting mechanical interlocking with the matrix material. These stronger bonding, in turn, potentially enhances load transfer and mechanical properties of the composite material. The polydopamine and nanoparticle treatment creates a structurally complex and chemically active interface on the carbon fibres, which translates into composites with enhanced mechanical properties and durability.

The fractured surface of CF/PPS composites after the ILSS test was observed by SEM to further elucidate the mechanisms in improving the fibre/matrix interfacial properties by the network of PDA/NPs. By comparing Fig. 11 (a) and Fig. 11 (b), as expected, the untreated carbon fibres could be completely separated from the PPS matrix in most areas on the surface due to relatively weak interfacial adhesion. After the introduction of the PDA/NPs network, a thick layer of PPS resin was found on the fractured surface. As shown in Fig. 11(b–c), in the absence of PDA/NPs network, the clean and smooth fibre bundles and gaps were captured along CF direction, which indicates that the crack propagates rapidly through the interface of CF/PPS. Fig. 11(e–f), however, shows that the rougher CF surface and zigzag patterns were observed. On one hand, the silica particles function as spacers, which could induce more smaller cracks in the cross-sectional area, instead of directly extending to the surfaces of carbon fibre. This contributed to the improved shear yield stress of the interface layer. The increase in impregnation percentage also leaded to the growing area of matrix residual in the separation process.

The comparative analysis of desized and treated carbon fibres via contact angle measurements is depicted Fig. 12. The contact angles were determined by depositing a water droplet onto the carbon fibre cloth, yielding a measurement of 122.97° for desized fibre and 33.82° for treated fibre respectively. This variation in contact angles indicates the wettability change induced by surface modifications. The elevated contact angle observed in the desized carbon fibre is indicative of a hydrophobic surface, which can be detrimental to the composite's mechanical integrity due to poor interfacial adhesion. In contrast, the treated carbon fibre significantly decreases contact angle, suggesting a transition to a more hydrophilic nature. This shift is primarily due to the introduction of polar groups on the carbon fibre surface, including hydroxyl groups from the silica nanoparticles and amine groups from the PDA layer.

Surface energy, governed by thermodynamic principles, influences the spreading and adhesion properties of a liquid on a solid substrate. A high contact angle is generally associated with low surface energy,

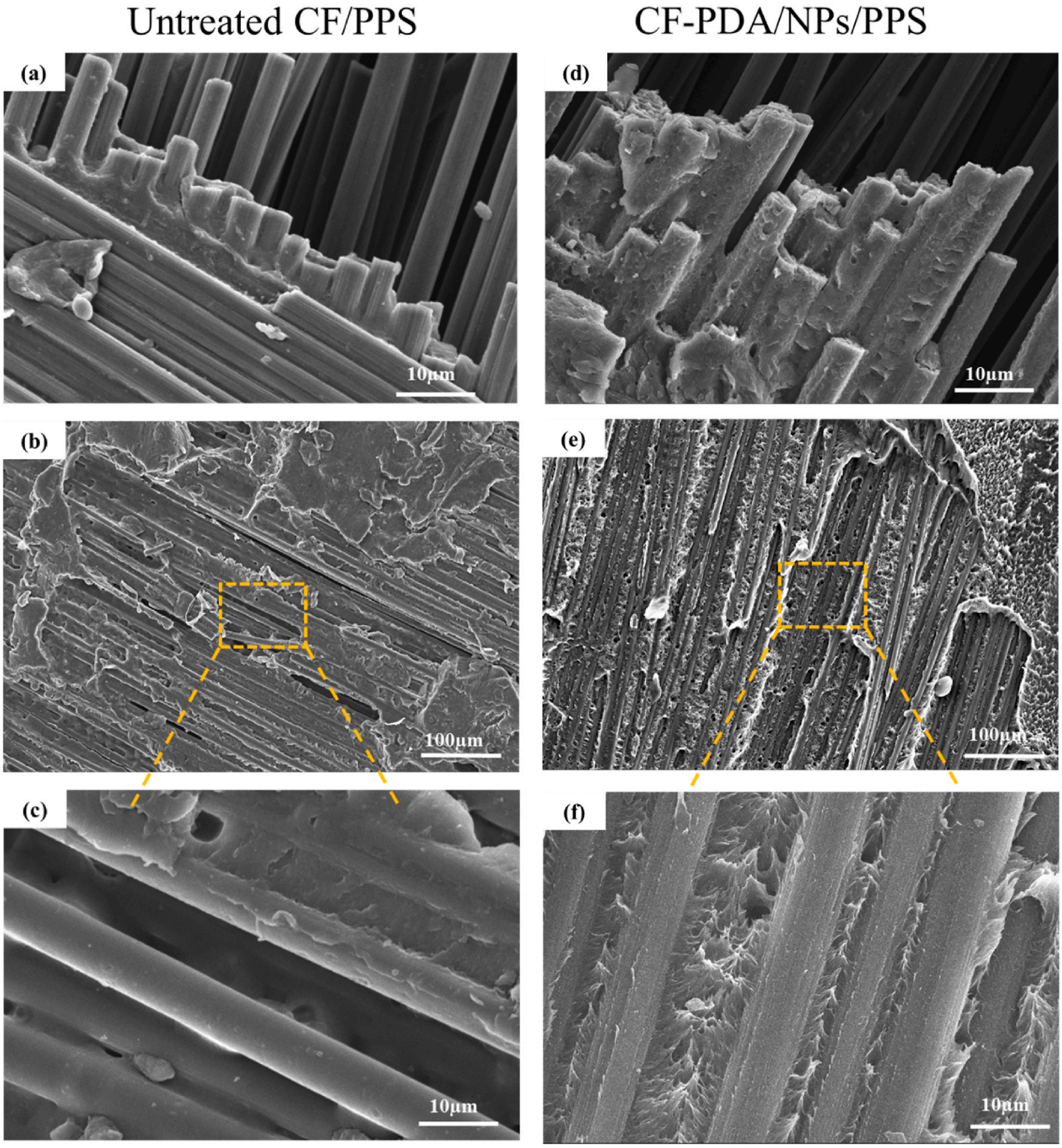


Fig. 11. Interlaminar shear fracture surface morphology of CF/PPS composites with untreated CF (a-x1000; b-x200; c-x1000) and CF-PDA/NPs (d-x1000; e-x200; f-x1000).

indicating a surface's limited adhesive potential (Packham, 2003). For carbon fibres, enhanced surface energy following treatment suggests improved wetting properties and the likelihood of stronger chemical bonding with the matrix material. Conversely, reduced surface energy implies lesser wetting and potentially weaker interfacial bonding. It can be concluded that the application of a PDA/NPs layer to desized carbon fibres not only improves wettability but also is likely to elevate the surface energy, thereby facilitating stronger interfacial adhesion within composites.

According to the above results, Fig. 13 presents the schematic diagram of the failure mode of CF/PPS composites for untreated CF and CF treated with PDA/NPs. Because of the formation of interactive network of the PDA-coated silica nanoparticles, the toughed interface layer around the CFs consisting of spherical shaped nanoparticles can dissipate the strain energy and transfer the stress to the interface region, which prevents the fibre-matrix de-bonding and therefore retard the propagation of cracks in the interlayer. As a result, the smooth fracture surface changes to a zigzag-like fracture surface with larger surface areas, thus leading to an enhanced ILSS strength.

4. Conclusions

Recycling and reuse of waste carbon fibres from end-of-life composites is a timely and important priority because of the detrimental environmental impact and landfill area shortage. This study demonstrates a one-step treatment that increases the ILSS of resulting CF/PPS composites by 28.4% through the establishment of a PDA/NPS network by the self-polymerisation of dopamine on the carbon fibre surface. The interfacial bonding between fibre and matrix exhibits great thermal stability in DMA results, which indicates that the network remains effective at service temperatures. Additionally, the network is proven to be reliable by thermal cycling, which indicates that this method could be

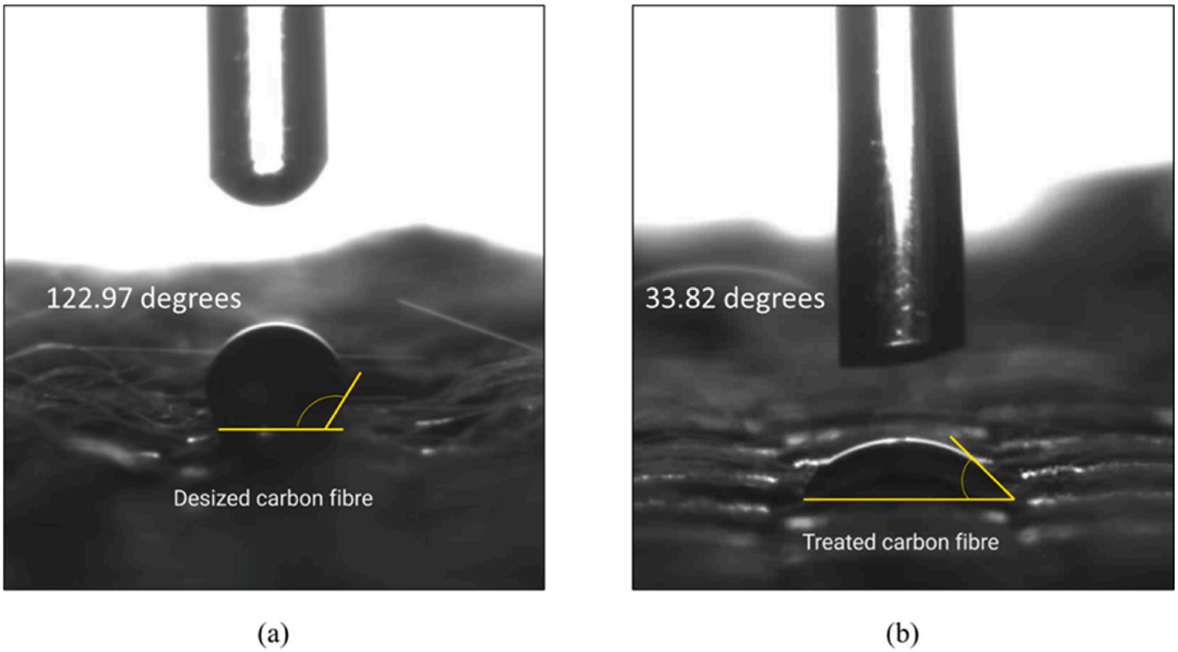


Fig. 12. Contact angle measurement of (a) desized carbon fibre cloth and (b) treated carbon fibre cloth.

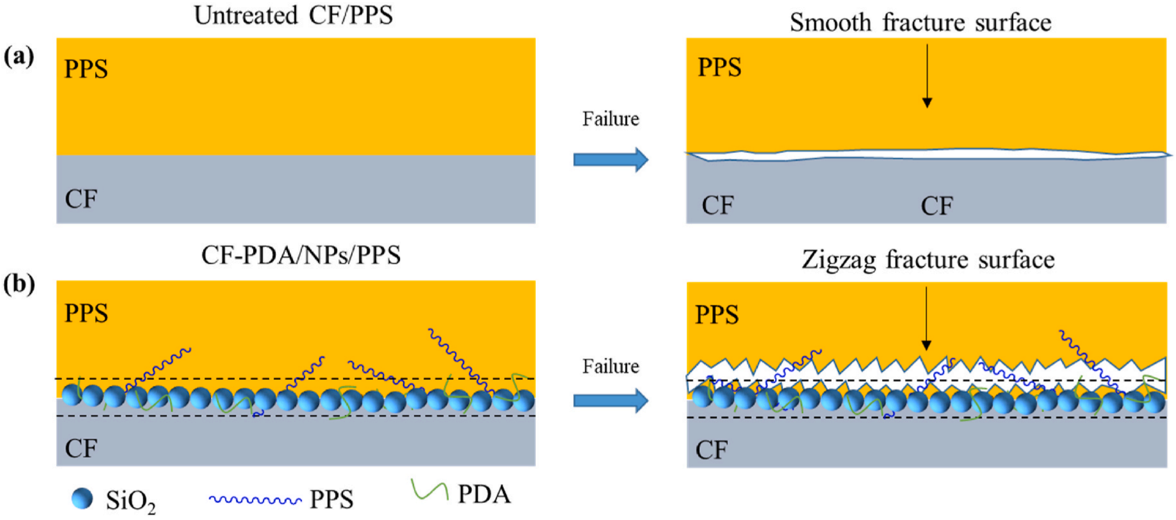


Fig. 13. Schematic diagram of the failure mode for CF/PPS composites: (a) untreated CF/PPS; (b) CF-PDA/NPs/PPS.

widely applied in complicated conditions. By grafting the polar functional groups on the carbon fibre surface, the roughness and wettability could be altered, leading to the improvement in fibre/matrix impregnation in the resulting composites. This process is free of toxic chemicals and easy to use, and thus constitutes a promising treatment for recycling and recovering carbon fibre for end-of-life advanced composites.

Compared to traditional wet chemical techniques that rely on organic solvents like chloroform and dichloromethane (Eyckens et al., 2021), or strong oxidizing agents such as hydrochloric acid (HCL) (Dong et al., 2020), nitric acid (HNO_3) (Hu et al., 2020), and sodium hydroxide (NaOH) (Li et al., 2022), our approach utilises the self-polymerisation of dopamine in an aqueous solution, avoiding the use of toxic chemicals. Notably, energy consumption was estimated to be reduced by 80% for our proposed reaction conducted at 60 °C for 2 h, followed by room temperature processing for 16 h, in contrast to reactions carried out at 220 °C for 6 h plus plasma treatment at 1.0 kW for 60 s (Dong et al., 2020). This significant reduction in energy requirement is projected to decrease CO_2 emissions in processing commercial recycled carbon fibre. Moreover, a cost reduction of 40% was estimated, considering both reactant and operational expenses. The proposed method aligns with key sustainability goals, including the minimisation of hazardous substances, resource efficiency, emissions reduction and development of sustainable materials.

While polydopamine is a robust, eco-friendly and versatile adhesive for treating carbon fibre to improve impregnation, its gentle oxidation and self-polymerisation process of its monomer, dopamine, can take nearly an entire day without the aid of additives or additional energy input, posing challenges in terms of efficiency and scalability of the coating process. Further research will explore the control of this oxidative polymerisation, possibly through the application of ultrasonic waves, addition of catalysts and/or increasing oxygen concentration, which could further accelerate the coating rate. Such acceleration could enable the automatic grafting of polydopamine/silica nanoparticles onto commercial recycled carbon fibre.

CRediT authorship contribution statement

Yahui Lyu: Writing – original draft, Visualization, Software, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation. **Vasileios Koutsos:** Writing – review & editing, Supervision. **Conchúr M. Ó Brádaigh:** Writing – review & editing, Supervision. **Dongmin Yang:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jclepro.2024.141855.

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