Carbon fiber reinforced epoxy composite properties improvement by incorporation of polydopamine sizing at fiber-matrix interface

Wenzhen Qin
Nanchang Hangkong University
Kaixuan Lei
Nanchang Hangkong University
Meiling Yan (Yanmeiling123@163.com)
Nanchang Hangkong University
Zhongkai Li
Nanchang Hangkong University
Yi Yan
Nanchang Hangkong University
Yongwei Hu
Nanchang Hangkong University
Zhijun Wu
Nanchang Hangkong University
Jianwei He
Nanchang Hangkong University
Liang Chen
Nanchang Hangkong University

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Abstract

To gain better interfacial and mechanical performances of carbon fiber reinforced epoxy resin (CF/EP) composites, the surface modification of CF was carried out via a simple mild method. The polydopamine (PDA) was applied to size the CF, as well as the influence of PDA sizing time on CF were elucidated. As the PDA deposited time increases, a growing number of PDA nanoparticles were adhered on CF surface, associating with appearance of PDA agglomerates. Through analyzing the results, the optimum PDA sized time was 24 h. The introduction of PDA increase the chemical reaction and mechanical interlocking between fiber and matrix. Compared with unsized CF, the tensile strength of PDA 24 h sized CF was improved by 27.0%, the interlaminar shear strength, flexural and impact strength of PDA 24 h sized CF/EP composites increased by 103.7%, 65.6% and 163.6%, respectively. Furthermore, the fracture topographies of CF/EP composite strikingly implied that the PDA has a positive impact on the mechanical performances of CF/EP composites.

1. Introduction

The carbon fiber reinforced epoxy resin (CF/EP) composites possess extensive promising application in aerospace, military, automobiles and sports fields, owning to their excellent mechanical properties and light weight [1, 2]. The CF/EP composites are compounded from reinforcements fiber, epoxy matrix and the interface between fiber and epoxy. Meanwhile, the interface acts as a bridge and link of stress transfer between the fiber and resin, which largely affects the properties of CF/EP [3, 4]. However, there is a weak interface in CF/EP composites due to the pristine carbon fiber surface is chemical inertia and smooth, which results in the premature failure of CF/EP and restricts the application [5–8]. Therefore, how to gain a good interphase in CF/EP composites is fundamentally important and interesting.

A plenty of CF surface modification approaches consisting of grafting active functional group and coarsening fiber surface have been recently discovered to heighten the interfacial bonding of CF/EP. The methods can be categorized into either oxidation process or sizing treatment. The techniques for fiber surface oxidation treatment include of grafting [9], plasma treatment [10], and electrochemical oxidation [11], which can introduce the functional groups and increase the fiber roughness. But, the above methods present several shortcomings of tedious process and costly, and the performances of the fiber itself may decline after surface oxidation treatment which influences the final performances of CF/EP.

Meanwhile, the sizing or coating treatment can enhance the roughness of fiber surface and introduce the interface layer, which advance the stress transfer to the CF [12–14]. More importantly, fiber sizing or coating is a convenient and effective method without any loss of fiber inner properties [15–17]. Recently, polydopamine (PDA) as a sizing agent for fiber surface modification is greatly appealing to researchers [18–21]. PDA coating exhibit outstanding adhesive properties and can stick to different material surfaces, such as metals, fibers, semiconductors, ceramics, glass, and even PTFE [22]. It has been reported that PDA layers are formed quickly through an oxidation-polymerization process of dopamine (DA) under moderate conditions [23]. Furthermore, PDA coating contain amino and catechol groups which can

associate with matrix to form chemical connections, hydrogen bonding and π-π bonding. Recently, many studies of improvement properties of composites by modification with PDA have been conducted [24–26]. Wu et al [27] proposed that the CF was modified by PDA, exhibiting the outstanding ability to heighten the interface strength of CF/EP composites. However, based on the PDA deposition time, the effects of PDA sizing modification on CF surface are rarely seen.

Therefore, in this study, CF was modified respectively with different PDA deposition time within 8h-32h through a simple sizing method. The process of CF-PDA and the possible formation mechanism of PDA are shown schematically in Fig. 1. The chemical, single fiber strength and morphology performances of the PDA sized CF were discussed. Furthermore, the interfacial and mechanical performances of CF/EP were assessed.

2. Experimental

2.1 Materials

Carbon fiber (T300B) was purchased from Japan Toray Company. Macklin Biochemical Technology Co., Ltd supplied Dopamine hydrochloride. The Tris (hydroxy methyl)-aminomethane was obtained from Shanghai Zhanyun Chemical Co., Ltd. Xilong Scientific Co., Ltd provided acetone and curing agent triethylene tetramine (TETA). Epoxy E51 was got from Jiangsu Sanmu Chemical Co., Ltd.

2.2 Synthesis of PDA coated CF

As-received CF was soaked in acetone for 48 h to eliminate the original sizing, which was named as rCF. The dopamine was firstly dissolved in a TRIS buffer solution (pH = 8.5), and the dopamine concentration was 2 g/L. Then the rCF was immersed in the above solution at room temperature. The fiber sized time were settled for 8, 16, 24, and 32 h, respectively. Finally, the sized CF were cleaned with distilled water and dried overnight. The corresponding sized fiber were designated as CF-PDA 8 h to CF-PDA 32 h.

2.3 Fabrication of CF reinforcement epoxy composites

The hand lay-up molding was employed to manufacture CF reinforced epoxy composites. In this process, a metal template was firstly wrapped with release paper. Then the CF bundle was tightly wound on the template to form one layer of CF. The E51 and curing agent TETA were added together with a ratio of 10:1 by weight and mixed well. Then the mixing epoxy was vacuumed for 30 min. After that, the mixing epoxy was uniformly spread on the CF. Five layers of CF with resin matrix was laid up and stacked fully. Finally, the CF/EP composites was placed on a hot press machine for curing which the curing condition was 10 MPa for 2 h at 120 ℃.

2.4 Characterize

Scanning electron microscopy (SEM, Nova NaNoSEM450) was applied to characterize the surface morphologies of CF. The compositions of the CF surface were analyzed by Fourier-transform infrared (FTIR, Shimadzu Company, IRPrestige-21). The content of PDA nanoparticles on CF and the thermal

stability of CF were investigated via Thermogravimetric (Diamond TG/DTA, Perkin Elme, USA), and the temperature was from 25 ℃ to 600 ℃ at the heating rate of 10 ℃/min under nitrogen atmosphere.

The monofilament tensile test was conducted on a XQ-1A fiber strength instrument (Shanghai Xinxian Instrument Co., Ltd.). The test rate was carried out at 1 mm/min following the GB/T 31290 – 2014. Each type fiber was performed 30 samples.

The interfacial properties of CF/EP composites were evaluated by interlaminar shear strength (ILSS). The 3-point short beam strength test was conducted to analyze the ILSS in accordance with the GB/T 3357 – 1982 standard. The test rate was at 1 mm/min. The dimensions of the ILSS composites samples were 20 mm in length, 6 mm in width, and 2 mm in thickness. At least 10 specimens of each composites were measured.

The flexural strength was measured via a three-point bending on a universal testing machine (WD-1, Shenzhen Suns Technology Stock Co., Ltd., Shenzhen, China) in accordance with GB/T 3357 – 1982. Impact tests were carried out using a pendulum impact testing machine (Shanghai laboratory instrument works Co. Ltd, Shanghai, China) at room temperature according to the QJ 1632-89 standard.

3. Results And Discussion

3.1 The CF surface morphology

The scanning electron microscope (SEM) was employed to discuss the surface morphology of CF before and after different sizing time of PDA, and the results are displayed in Fig. 2. A relatively clean surface and some grooves are noticed for untreated carbon fiber (rCF) (Fig. 2a). These grooves are produced during preparing CF. However, the surfaces of PDA sized CF (CF-PDA) differ in rCF. It can be seen exactly that the density of PDA nanoparticles rises remarkably along with deposition time. A fairly small amount PDA nanoparticle is attached on the CF surface when deposited time of PDA is 8 h (Fig. 2b). While, a thin PDA layer and some PDA nanoparticles appears on the CF when the deposition time of PDA increased to 16 h (Fig. 2c). Figure 2d shows that when the deposition time rise to 24 h, PDA nanoparticles are distributed uniformly on the CF. This is in favor of raising the surface activity which help to boost the interphase interaction between fiber and matrix. What's more, it can be seen from the Fig. 2e that after PDA depositing to 32 h, large amounts of PDA nanoparticles are settled on the fiber surface. Meanwhile, some large PDA nanoparticles and aggregates are appeared on the CF surface which might impact on the mechanical performances of CF/EP.

3.2 The tensile strength of monofilament

The strength of monofilament is applied to describe fiber intrinsic strength, which influences the overall mechanical performances of CFPR [28]. The single fiber tensile experiments were performed for evaluating how that the impact of PDA sizing on tensile strength of monofilament, and the results are revealed in Fig. 2f. The single fiber tensile strength of rCF is 2.82 GPa. For PDA sized CF, the tensile

strength first increases and then declines with the increase of PDA deposition time, but all the tensile strengths of PDA sized CF are higher than rCF. It was noticeable that the tensile strength of monofilament is excellent when the treated time is 24 h, and the tensile strength is 3.58 GPa which goes up by 27.0% compared to rCF. The result demonstrates that evenly distributed PDA nanoparticles repair the surface faults of fiber during the production process and protect the bulk structure of CF, which is profit to enhance the tensile strength by decreasing the concentration of the stress [23]. Besides, there has the extra resistance providing by the PAD nanoparticles on CF to resist the loading force, which also raise the tensile strength [29]. However, when the deposition time of PDA increases to 32 h, the single CF displays a decrease in tensile strength. Presumably it's because that the decrease of single fiber tensile strength is caused by stress concentration on the aggregates of PDA nanoparticles on fiber surface as exhibited in Fig. 2e, which might enhance the crack initiation and propagation [30].

3.3 Surface chemical structures of CF

The FTIR was applied to determine the chemical structures of rCF and CF-PDA were, and the results are displayed in Fig. 3a. It's clear to see that new bands are appeared in the spectrum of PDA sized CF in comparison to rCF. For PDA sized CF, the new peaks at 3424 cm^{-1} and 3200 cm^{-1} are put down to the O-H and N-H bonds stretching vibration, which is ascribed to the PDA [21], respectively. Meanwhile, the new double peaks appeared at 2924 cm^{-1} and 2856 cm^{-1} are associated with C-H bonds stretching vibrations. The peaks at 1625 cm^{-1} result from the stretching vibration of C = C bond in the indole aromatic ring of PDA [30]. The new band at 1499 cm^{-1} corresponds to the shearing vibration of N-H [31]. The peak at 1394 cm^{-1} represents the stretching vibration of and 1625 bond [32]. And, the peaks at 1243 cm^{-1} and 1056 cm^{-1} both derive from shearing vibration of C-O bonds [33]. The FTIR results further evidence that PDA nanoparticles are successfully coated on CF.

In the interests of assessing the effect of PDA coating on the thermal performances of CF, the TGA measurement was put into effect from 25 to 600 ^{o}C in nitrogen atmosphere. Figure 3b. shows the TGA results. It can be observed that there is almost no mass decrease of rCF with the temperature rising. This signifies the excellent thermal stability of CF. However, the weight loss of PDA sized CF is 3.1%, which imply that PDA nanoparticles are successfully deposited on CF surface. Meanwhile, the major weight decrease of PDA sized CF commences at approximately 301 ^{o}C, which matches published initiation decomposition temperature of PDA [19].

3.4 Performances of CF/ epoxy composites

The ILSS is a significant method to evaluate the interfacial performances of CF/EP composites. The ILSS of rCF/EP composites and PDA sized CF/EP composites at different PDA deposition time are displayed in Fig. 4a. As demonstrated in the result, all the ILSS of PDA sized CF/EP composites are higher than rCF/EP composites. The peak ILSS value appears in PDA 24 h sizing. A significant improvement of 103.7% is achieved in the ILSS of PDA 24 h sized CF/EP composites compared with that of rCF/EP composites. The enhancement of ILSS are as a result of increasing the CF surface wettability and compatibility by PDA nanoparticles, which is profit to heighten the mechanical occlusion between fiber

and matrix. Moreover, the successful deposition of PDA nanoparticles brought into aplenty of amino and hydroxyl groups on the PDA sized CF surface, which could also enhance the surface reaction activity and promote the chemical bonding in the interface of CF/EP composites [24]. However, the ILSS of PDA 32 h CF/EP composites is below than that of PDA 24 h CF/EP composites. The aggregates of PDA on the surface of CF could result in partial stress concentration and raise the chances of crack initiation and propagation, which causes the decease of ILSS [34].

The flexural and impact strength were applied to examine the mechanical performances of CF/EP composites, and the results are demonstrated in Fig. 4b and c. The flexural and impact strength of rCF/EP composites are 292.1 MPa and 47.6 kJ/m^2, respectively. After sizing by PDA, the two strengths of all CF/EP composites are enhanced. Among them, the flexural and impact strength of PDA 24 h CF/EP composites present maximal value of 483.6 MPa and 125.3 kJ/m^2, respectively, which are increased by 65.6% and 163.6% in comparison of rCF/EP composites, respectively. The remarkable improvement of PDA 24 h sized CF/EP composites are attributed to the following reasons. First, well-distributed PDA on CF surface repair the defects of CF and decrease the stress concentration, which is benefit to strengthen the mechanical properties of CF/EP composites. Second, the PDA nanoparticles heightens the interfacial bonding in fiber/matrix, stimulating the efficiency of stress transfer in CF/EP composites, which the enhancement of mechanical properties.

The diagrams of failure mechanism and the morphologies of failure surface of CFRP are displayed in Fig. 5. For rCF/EP composites, almost no epoxy matrix is remained on the fiber surface and the fiber surface is relative clean (Fig. 5a), revealing that the fracture mechanism is interface deboning. Meanwhile, some holes are clearly appeared on the fracture image which the CFs are pulled out of the matrix, indicating poor interfacial bonding. In contrast, for PDA sized CF/EP composites, as showed in Fig. 5b, epoxy matrix residue adhered to the CF surface and few CFs are extracted, revealing that the fracture mechanism of PDA sized CF/EP composites is an integration of fracture and interface deboning [35]. The findings indicate that strong fiber/matrix adhesion is strong in PDA sized CF/EP composites. The enhancement of interfacial properties in PDA sized CF/EP composites can be summed up to the following reasons (as shown in Fig. 5c). First, the amino groups of PDA sized CF can be attacked and reacted with epoxide group, which increases the bond strength between PAD sized CF and epoxy matrix. Second, the catechol groups on the PDA sized CF can associate with hydroxyl groups due to ring-opening reaction of epoxy to form hydrogen bonding. Moreover, based on the graphite microcrystals of CF, the aromatic ring structure of PDA and epoxy matrix, there may form π-π stacking interaction among CF, PDA and epoxy matrix. Meanwhile, the rough surfaces of PDA sized CF enlarge the contact area between fiber and matrix, which enhances the mechanical interlocking in interface of PDA sized CF/EP composites. Therefore, PDA is acted as a bridge linking the fiber and matrix, increasing the interface and mechanical properties of CF/EP composites.

4. Conclusion

In this study, the surface of CF was modified by polydopamine, and the effect of deposition time by PDA on the interfacial and mechanical properties of CF reinforced epoxy composites was analyzed. The results of SEM, FTIR and TGA indicated that the PDA was successfully deposited on the CF surface. The SEM images showed that the density of PDA on the surface of CF increase in proportion to the PDA sized time. However, some PDA aggregates were observed with the sized time increasing. The uniform PDA nanoparticles may repair the surface faults of CF when the sized time was 24 h, which resulted in the tensile strength of monofilament. The ILSS, flexural and impact strength of PDA 24 h sized CF/EP composites were enhanced by 103.7%, 65.6% and 163.6%, respectively. The increasing surface roughness, compatibility and functional reaction groups of CF by PDA nanoparticles, which is profit to increase the mechanical occlusion and chemical bonding in the interface between CF and epoxy.

Declarations

Credit authorship contribution statement

Wenzhen Qin: Conceptualization, Writing-review. Meiling Yan and Zhongkai Li: Data curation, Review and editing. Kaixuan Lei, Yongwei Hu, Zhijun Wu and Jianwei He: designed and conduct experiments. Yi Yan and Chen Liang: Validation.

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Conflict of interest The authors declare no competing interests.

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Figures

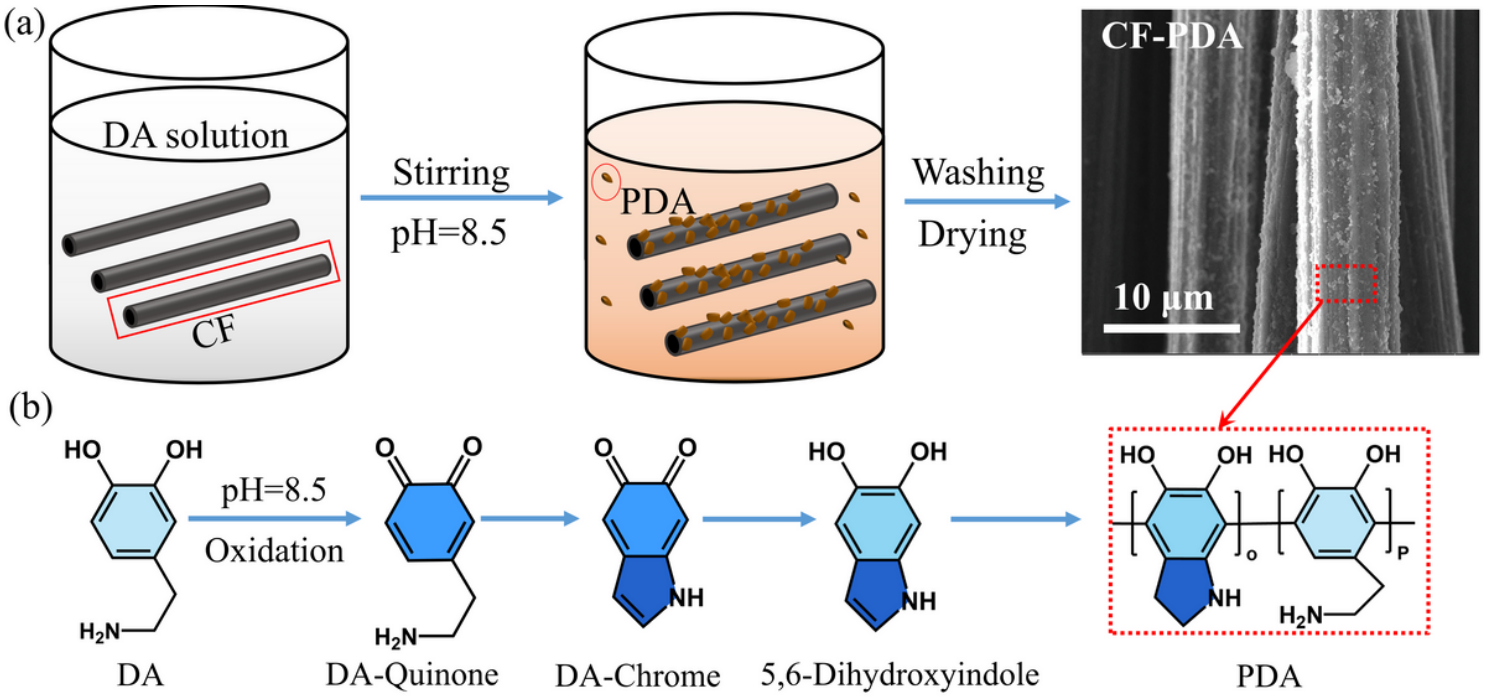

Figure 1

a Synthesis of PDA-CF, **b** Possible formation mechanism of polydopamine

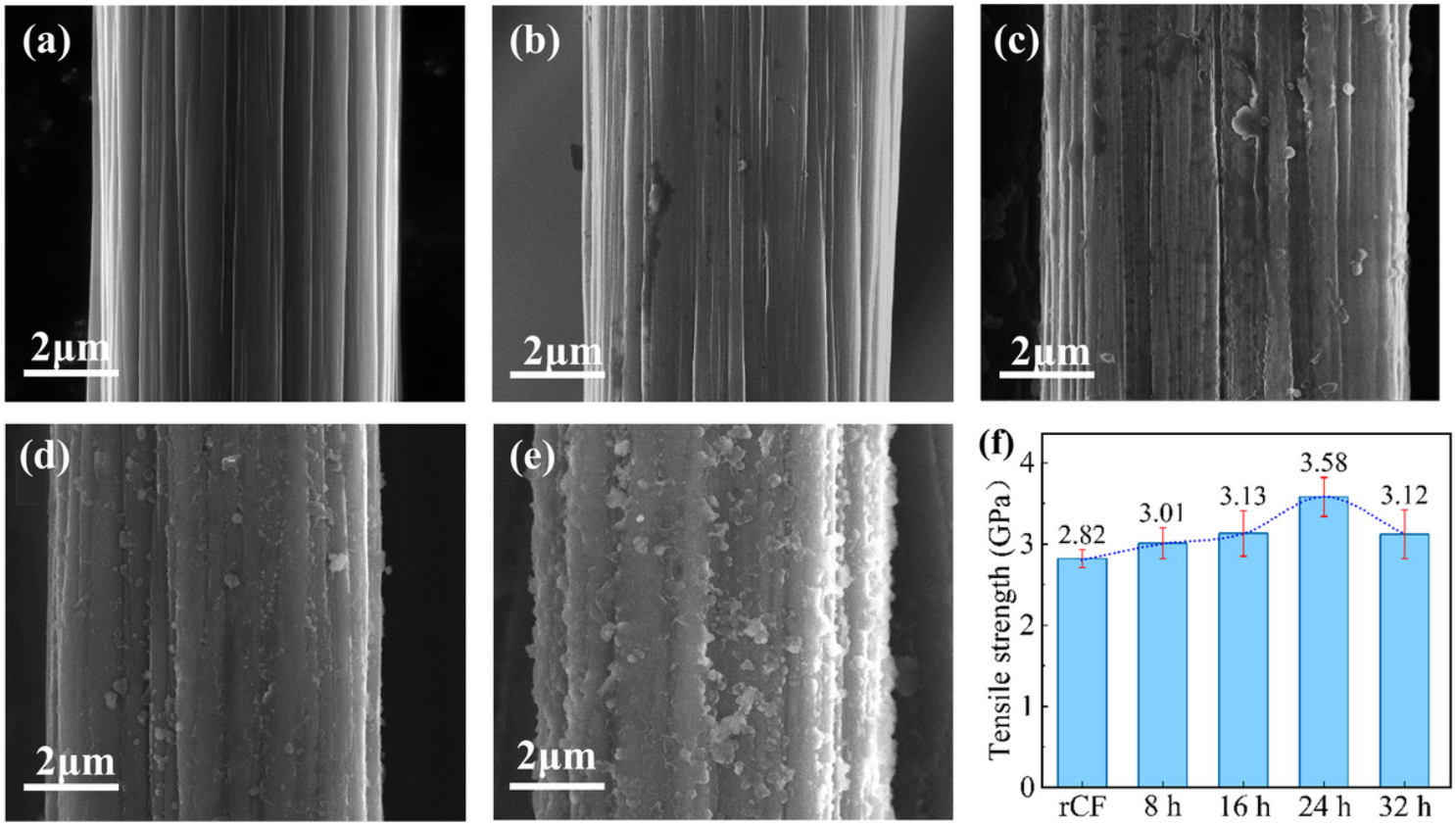

Figure 2

SEM images: **a** rCF, **b** CF-PDA 8 h, **c** CF-PDA 16 h, **d** CF-PDA 24 h, **e** CF-PDA 32 h. **f** the tensile strength of monofilament

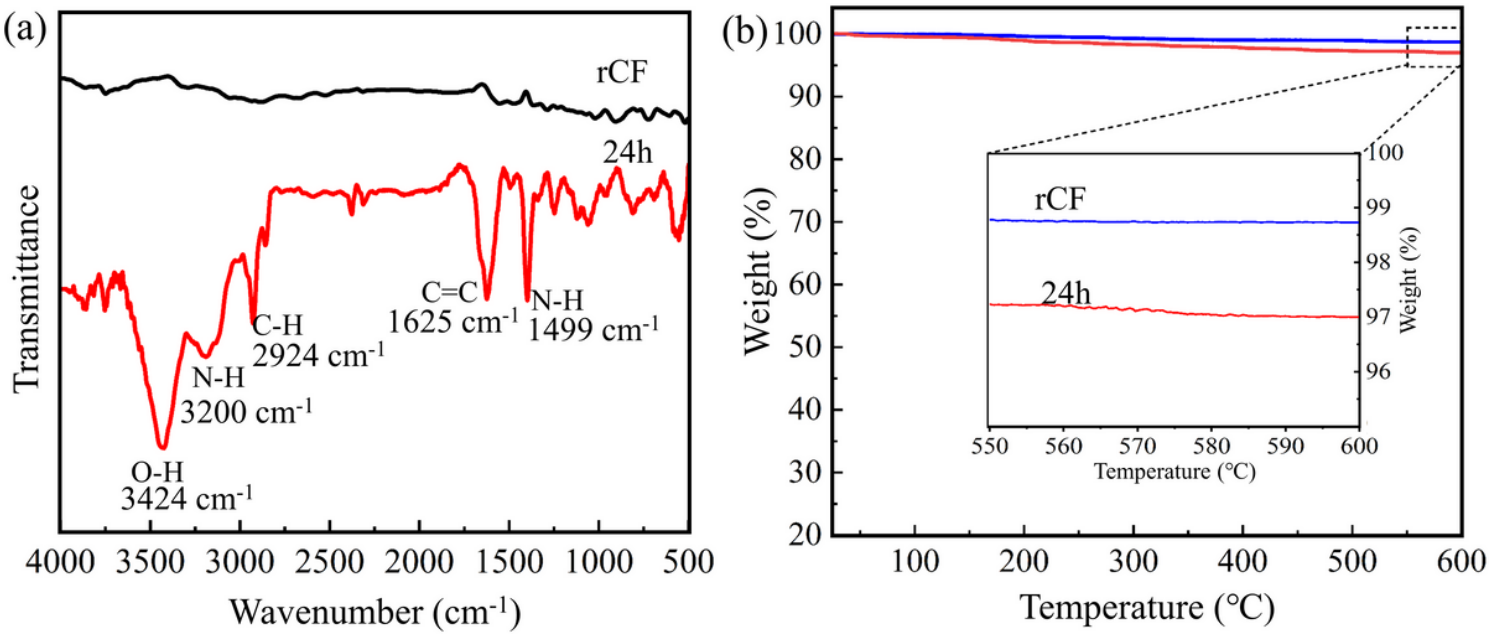

Figure 3

a Infrared spectrum and **b** the TGA curves of rCF and CF-PDA 24 h

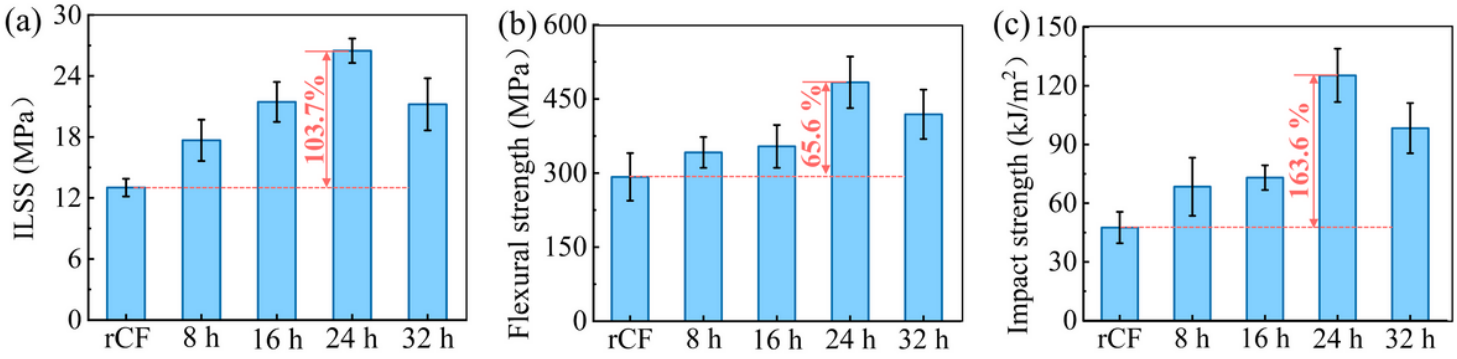

Figure 4

PDA sized CF/EP composites with different sizing time: **a** ILSS, **b** flexural and **c** impact strength

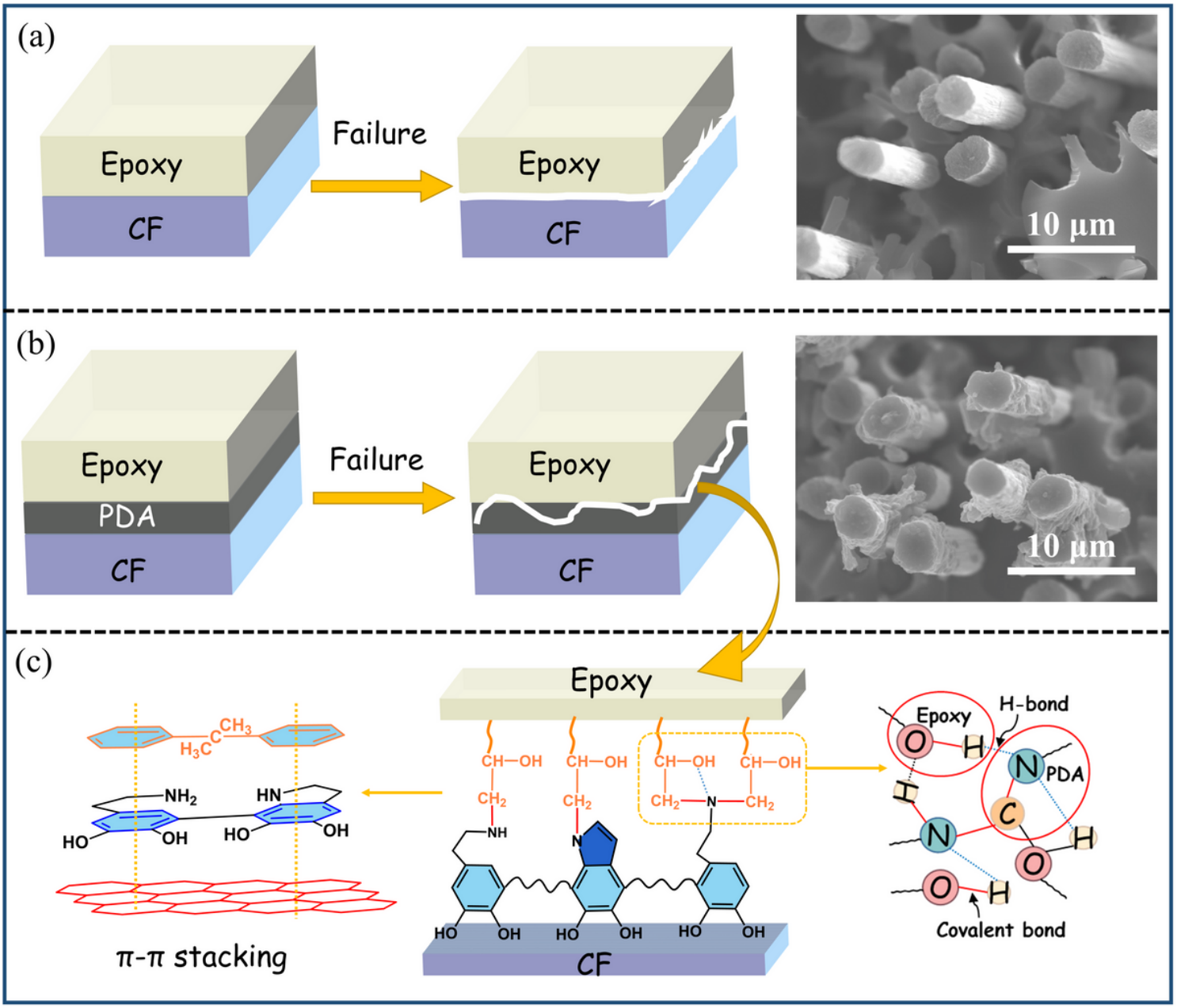

Figure 5

Schematic diagrams and morphologies of the failure of composites interface of **a** rCF/EP and **b** PDA sized CF/EP. **c** reaction mechanism between PDA on CF and EP matrix in interface