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Effect of Surface Modification on Properties of Kevlar/Epoxy Polymer Composites

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Abstract

In this study, a high-performance Kevlar/epoxy polymer composite was prepared using a hand lay-up technique. The Kevlar fiber was treated with calcium chloride/ethanol solution ($CaCl_2$/EtOH) to enhance the interfacial interaction between the fiber and the epoxy matrix. The optimization of fiber surface treatment and fiber content for the best tensile strength (TS) of Kevlar/epoxy polymer composite was achieved using Box-Behnken design (BBD) through response surface methodology (RSM). Three parameters: $CaCl_2$ concentration (A), $CaCl_2$ treatment time (B), and Kevlar content (C) were chosen. The results indicated that the C factor had the most effect on the response and the B factor was the least important factor. The optimal parameters extracted by Design Expert Software were $CaCl_2$ concentration of 6.59 %, $CaCl_2$ treatment time of 5.58 h, and Kevlar fiber content of 42%. Under these conditions, the TS of the polymer composite obtained experimentally was 409.6 MPa, which was not significantly different from the predicted TS value of 408.7 MPa. The surface modification of Kevlar fiber causing higher roughness due to the complex formation between $CaCl_2$ and -C=O group has significantly improved the split tear strength (Ds) of the Kevlar/epoxy polymer composite to 3.1 N/mm, compared to untreated Kevlar fiber of 2.4 N/mm.

Keywords: Kevlar fiber, surface modification, epoxy composite, $CaCl_2$ treatment, Box–Behnken design.

INTRODUCTION

High-performance polymer composites have attracted much research interest for decades because these materials have applications in high-tech sectors such as aerospace, aviation, and automobile [1–3]. In general, the mechanical properties of polymers depend strongly on the polymer matrix, the reinforcement materials, the composition, the interaction matrix and reinforcement, and the processing parameters like curing temperature, curing time, and curing pressure [4–8]. One widely used reinforcement material for advanced polymer composites is Kevlar fiber – an aromatic polyamide fiber – due to its excellent properties, such as superior tensile strength and modulus, lightweight, thermal stability, and impact resistance [7, 9–11]. However, the surface of Kevlar fibers is chemically inert and smooth which is derived from the high crystallinity of the fiber surface layer and the lack of polar functional groups in the polymer molecular chain. Thus, it is difficult to form strong bonds

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between Kevlar and epoxy through chemical and physical interaction, often resulting in poor adhesion between Kevlar fiber and the resin matrix [11]. To improve the interface interaction between the reinforcing fiber and the polymer matrix, different strategies can be applied, e.g. mechanical interlocking, polarity matching, and chemical bonding [12, 13].

Various approaches to surface modification for Kevlar fibers have been developed, including chemical, physical techniques, and a combination of the two methods [1, 7, 12–16]. Conventional chemical approach, for example, a graft reaction in the active hydrogen atoms of the Imide group, has a poor modification efficiency because the hydrogen atoms of the Imide groups had been inactivated intensively due to hydrogen bonding among Imide groups, carbonyl group, and phenyl ring, along with the steric hindrance effect of phenyl rings [12, 17].

To improve fiber-matrix interfacial bonding in Kevlar/epoxy composite laminates, Ramasamy N et al. pre-treated Kevlar fiber with phosphoric acid (PA) followed by epichlorohydrin (ECH) treatment. The results indicated an increase of 3.7% tensile modulus and 42.5% flexural modulus comparable to the non-pre-treatment ECH surface modification [18]. Ultrasonic treatment of aramid fiber surface increased by 13% of the interlaminar shear strength of aramid/epoxy composite without reducing the tensile strength [19]. Plasma treatments of aramid fiber in order to improve the mechanical properties of aramid fiber reinforced polymer composites have been also reported in several papers [1, 11, 20, 21]. The ultrasonic or plasma techniques are environment friendly, but it normally needs high-cost equipments.

One of the simple and low-cost methods is metal ion etching on the surface of aramid fiber using Lewis acids [17, 22–28]. By this method, the separation of polyamide 66 from a mixture of cellulose fibers with calcium-ethanol-water has been successfully investigated by our group [17]. Metal ion etching on the fiber' surface is probably due to alcoholysis as follows: the complexation of calcium ions with the carbonyl groups causes the secondary amine to remove protons, increasing the positive charge of carbonyl carbon and promote the alcoholysis of the amide [22–28]. Figure 1 presents the possible metal ion complexation reaction of $CaCl_2$ with Kevlar fiber [23].

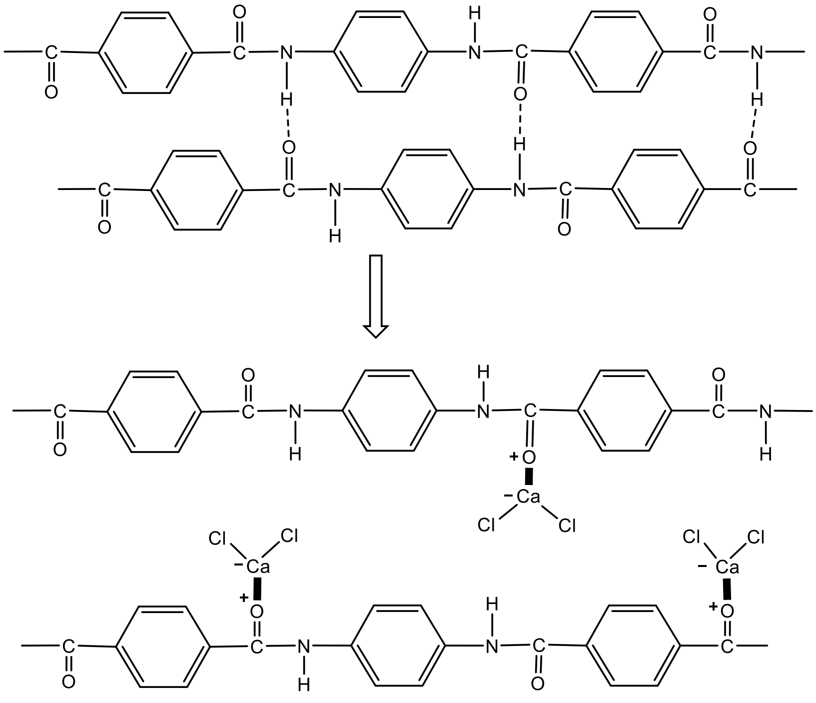

Figure 1. Illustration of complexation reaction of $CaCl_2$ with kevlar fiber [23].

By treating with $CaCl_2$/EtOH solution, hydrogen bonding in the Kevlar structure is disrupted, resulting in solubilization of the polymer chain and reduced crystallinity. This leads to the formation of voids and pores and therefore increased roughness of the fiber surface. The surface modification of Kevlar could increase the interaction between Kevlar fiber and the polymer matrix [23]. The $CaCl_2$ treatment of Kevlar fiber is also accomplished with the decrease of the mechanical strength of the Kevlar fiber [24]. Thus, an optimization of the fiber surface treatment is necessary to achieve a good balance between interface interaction and mechanical properties of Kevlar-reinforced polymer composites.

Until now, the Box-Behnken design through response surface methodology is still considered to be more proficient and powerful than other design methods such as central composite design, three level full factorial design etc [29]. The Box-Behnken design (BBD) was successfully used to optimize the mechanical properties of poly lactic acid composite [30].

In this work, a high-performance Kevlar/epoxy polymer composite was prepared by hand lay-up technique. To obtain the highest tensile strength of the Kevlar/epoxy polymer composites, the optimization of the treatment of Kevlar fiber with $CaCl_2$/EtOH solution and fiber content was conducted using Box-Behnken Design through response surface methodology. The structure and properties of the polymer composite were characterized by Fourier-transform infrared spectroscopy (FT-IR), atomic forced microscopy (AFM), scanning electron microscopy (SEM), X-ray diffraction (XRD), tensile testing, and split tear strength measurement.

MATERIALS AND METHODS

Materials

Kevlar fabric 400D was purchased from HY Network Co. Ltd (Shanghai, China) (weight 120 g/m^2, thickness: 0.19 mm). Epoxy resin - EPICOTE™ Resin MGS® LR 385- with an epoxy value of 0.58 ÷0.64 (equivalent/100g), density of 1.16÷1.20 (g/cm^3) and Hardener LH 385 (density 0.95÷0.99, amine value of 480÷550 (mg KOH/g) was obtained HEXION company, Germany. Calcium chloride anhydrous was purchased from Shanghai Macklin Biochemical Co., Ltd, China. Ethanol and acetone were received from Sigma-Aldrich, Singapore. All chemicals were analytical grade and used without further purification.

Surface Modification of Kevlar Fiber and Preparation of Kevlar/Epoxy Polymer Composites

Before surface modification, the Kevlar fabric was cut into squares (15cm×15cm), then immersed in acetone, followed by washing with distilled water to eliminate impurities on the surface of the fiber [24]. Then it was dried in a vacuum oven at 60ºC for 4 h.

The surface modification of Kevlar by $CaCl_2$/EtOH solution was carried out according to the references [23,24]. From the results in previous papers [23,24]. different $CaCl_2$/EtOH solutions with concentrations of 1; 5.5; 10 % (w/v), respectively were prepared. The cleaned Kevlar samples were put in a roll-form into a 3-mouth flask equipped with a reflux condenser and $CaCl_2$/EtOH solution was added. Subsequently, the system was heated to 80ºC (the boiling temperature of EtOH) in an oil bath. The treatment times of Kevlar fiber were 1, 5.5, and 10 h, respectively. Afterward, the treated Kevlar fabrics were washed with acetone and distilled water to remove any unreacted agents and then dried in a vacuum oven at room temperature for 24 h.

Kevlar/epoxy polymer composite was fabricated using a hand lay-up technique with 5 plies of Kevlar fabrics [5, 31]. Figure 2 shows the preparation procedures of Kevlar/epoxy composites.

Based on single screening experiments (data not shown), the Kevlar content in polymer composites was chosen in the range of 35÷45 %. The hardener: the epoxy ratio was fixed at 35:100 (w/w). Epoxy and hardener were mixed for five minutes using a magnetic stirrer and degassed in a vacuum chamber at room temperature for 10 min before use. The Kevlar/epoxy polymer composites were cured at room temperature (RT) for 24 h at a pressure of 20 kg/cm^2 by using a hydraulic pressure machine. Afterward, the Kevlar /epoxy polymer composites were post-cured at 50ºC for 15 h in an oven.

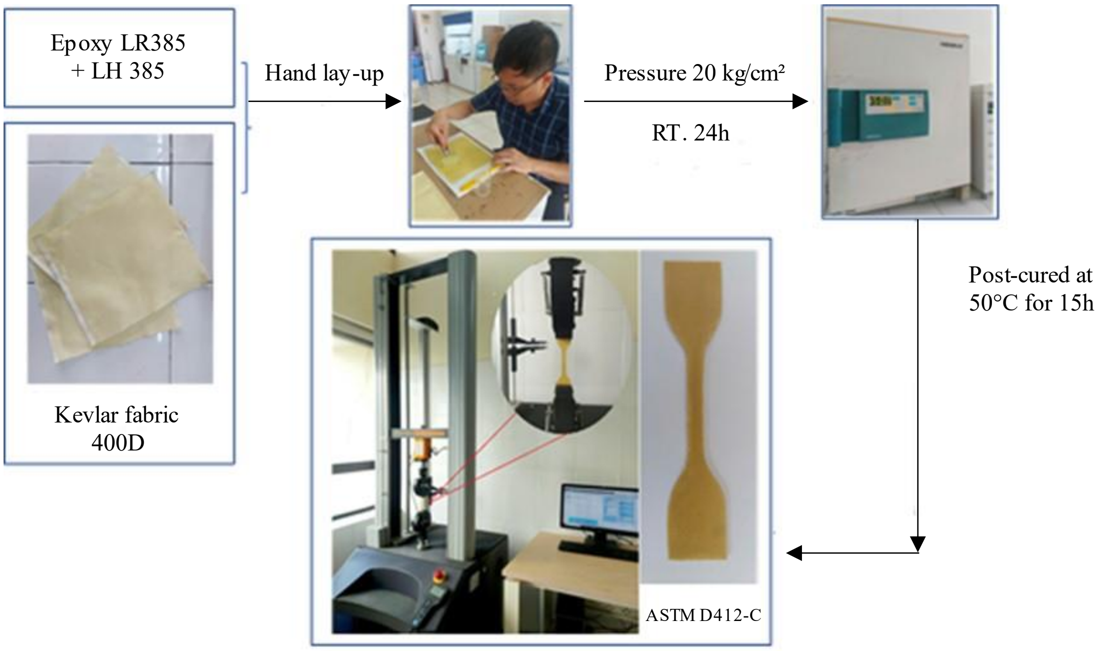

Figure 2. Preparation procedures of kevlar/epoxy composites.

The optimization of the surface treatment of Kevlar fiber and the fiber content was carried out using the response surface methodology (RSM) with a three-level Box-Behnken design to obtain the highest TS of Kevlar/epoxy polymer composites. The three most important parameters that were chosen for the optimization were $CaCl_2$ concentration (A), $CaCl_2$ treatment time of Kevlar fiber (B), and Kevlar content (C). A total of 15 experiments were performed. The code variables are shown in Table 1.

Table 1. Independent variables and their level.

Independent variables	**Level**		
	-1	***0***	***+1***
A: $CaCl_2$ concentration (%)	1	5.5	10
B: $CaCl_2$ treatment time (h)	1	5.5	10
C: Kevlar content (%)	35	40	45

Three experimental replicates of each case were performed and the average values have been expressed for the experimental responses.

The behavior of the system was explained by the following quadratic equation:

$$TS = b_0 + b_1A + b_2B + b_3C + b_1b_2AB + b_1b_3AC + b_2b_3BC + b_1A^2 + b_2B^2 + b_3C^2 \quad (1)$$

Whereas: TS is tensile strength; A, B, and C are independent variables; b_0 is the off-set term, b_1, b_2, b_3, b_1b_2, b_1b_3, and b_2b_3 are coefficients estimated by the model.

Characterization Methods

ATR-FTIR spectra of the samples were collected using a Nicolet Nexus 760 FT-IR spectrometer using attenuated total reflectance (ATR) technique.

XRD analysis: The crystal structure of Kevlar fibers was determined by XRD instrument using D8 ADVANCE X-Ray (Bruker) with a 2-theta range from 10 to 80° using CuKα radiation. The crystallinity degree (X_{CR}) was calculated as the ratio of deconvoluted diffraction peak areas to the total scatter under the normalized intensity scan by the equation [23].

$$X_{CR} = \frac{\sum_{i=1}^{B} \int_{2\theta_1}^{2\theta_2} I_i(2\theta)d(2\theta)}{\int_{2\theta_1}^{2\theta_2} I_T(2\theta)d(2\theta)} \qquad (2)$$

The surface morphology of Kevlar fibers was analyzed by atomic force microscopy (AFM) (MFP-3D Infinity Asylum Research AFM, Oxford Instruments), using the tapping mode. The roughness of the Kevlar surface was characterized by mean square roughness (Rq) and arithmetic mean roughness (Ra) calculated automatically by the software.

Scanning electron microscopy (SEM) of the sample were taken using SEM JSM-7500F, JEOL (Japan).

The tensile strength of the polymer composite was measured on a universal testing machine (Gotech Testing Machines (see Figure 2) according to the ASTM D3039 standard with the crosshead speed of 2 mm/min at room temperature. The tensile testing specimens was made according to the ASTM D412-C (see Figure 2).

The split tear strength (Ds) of the Kevlar/epoxy polymer composites was measured according to the ASTM D470 standard. The dimension of the sample (length x width) was 100 x 20 (mm).

Analysis of the experimental design and calculation of predicted data were carried out using Design Expert Software (version 12.0).

RESULTS AND DISCUSSION

Parameter Optimization By Response Surface Methodology

Box–Behnken design (BBD) with a total number of 15 experiments was performed to optimize the three individual parameters. The process variables under Box-Behnken design conditions and experimental data are shown in Table 2.

As indicated in Table 2, the tensile strength of the Kevlar/epoxy polymer composite depends strongly on the surface modification of Kevlar fibers as well as Kevlar content and ranges from 321.1 to 404.8 MPa. At longer treatment time and higher $CaCl_2$ concentration the surface structure of Kevlar fiber had been destroyed, and the strength of the Kevlar fiber was reduced [23, 24]. It is expected that, at optimal conditions, strong mechanical bonding between epoxy and Kevlar fibers due to filling of the pits, notches and grooves by epoxy, at the same time, Kevlar still also maintained its mechanical properties without significant change. Long time treatment at EtOH boiling temperature may induce large number of these pits and grooves on the fiber surface forming defects and stress concentration areas that reducing the mechanical properties of the polymer composite

The analysis of variance (ANOVA) was used to check the statistically significant of the model and the factors in the fitted model for the tensile strength of the composite (Table 3). The Model F-value of 1404.51 with the p-value <0.0001 suggested that the model is significant. The lack-of-fit of F-value 8.45 with p-value 0.1706 was insignificant, which indicated that the model was fitted to all data. The quadratic model was statistically significant for the response and it could be used for further studies. The high coefficient R^2 of 99.9% indicated that 99.9 % of the variability in the response of tensile strength can be explained by the model equation (3), adjusted R^2 was 99.9%, predicted R^2 was 99.4 % indicating that the model was well adapted to the response.

Table 2. Box-behnken experimental design with the independent variables.

No	A: $CaCl_2$ concentration (%)	B:treatment time (h)	C: Kevlar content (%)	Tensile strength (MPa)
1	-	-	0	335.8
2	+	-	0	370.6
3	-	+	0	362.5
4	+	+	0	367.6
5	-	0	-	321.1
6	+	0	-	340.6
7	-	0	+	359.3
8	+	0	+	385.2
9	0	-	-	325.8
10	0	+	-	333.2
11	0	-	+	363.2
12	0	+	+	378.1
13	0	0	0	403.4
14	0	0	0	404.2
15	0	0	0	403.8

The P-value was used to check the significance of each coefficient. P-values less than 0.05 indicate model terms are significant. The smaller the P-value was, the more significant the corresponding coefficient was. Both the linear and quadratic terms of $CaCl_2$ concentration (A), treatment time (B), and Kevlar content (C) had a p-value < 0.0001, in which the C factor had the most effect on the response and the B factor was the least important factors. All the interaction terms A×B, A×C, and B×C influenced the response TS ($p<0.05$), in which the interaction term A×B had the most influence on tensile strength.

The following quadratic model explains the experimental data:

$$TS = 403.8 + 6.67\,A + 4.21\,B + 7.99\,C - 2.95\,AB + 4.40\,AC + 3.82\,BC - 6.0\,A^2 - 5.73\,B^2 - 14.08\,C^2 \qquad (3)$$

Table 3. ANOVA for quadratic mode.

Source	Sum of squares	df	Mean square	F-value	p-value	
Model	11066.81	9	1229.65	1404.51	< 0.0001	significant
A-$CaCl_2$ concentration	909.51	1	909.51	1038.85	< 0.0001	
B-treatment time	264.50	1	264.50	302.11	< 0.0001	
C-Kevlar content	3407.25	1	3407.25	3891.78	< 0.0001	
AB	220.52	1	220.52	251.88	< 0.0001	
AC	10.24	1	10.24	11.70	0.0188	
BC	14.06	1	14.06	16.06	0.0102	
A^2	1722.68	1	1722.68	1967.66	< 0.0001	
B^2	1965.99	1	1965.99	2245.56	< 0.0001	
C^2	3468.64	1	3468.64	3961.89	< 0.0001	
Residual	4.38	5	0.8755			
Lack of Fit	4.06	3	1.35	8.45	0.1076	not significant
Pure Error	0.3200	2	0.1600			
Cor Total	11071.19	14				

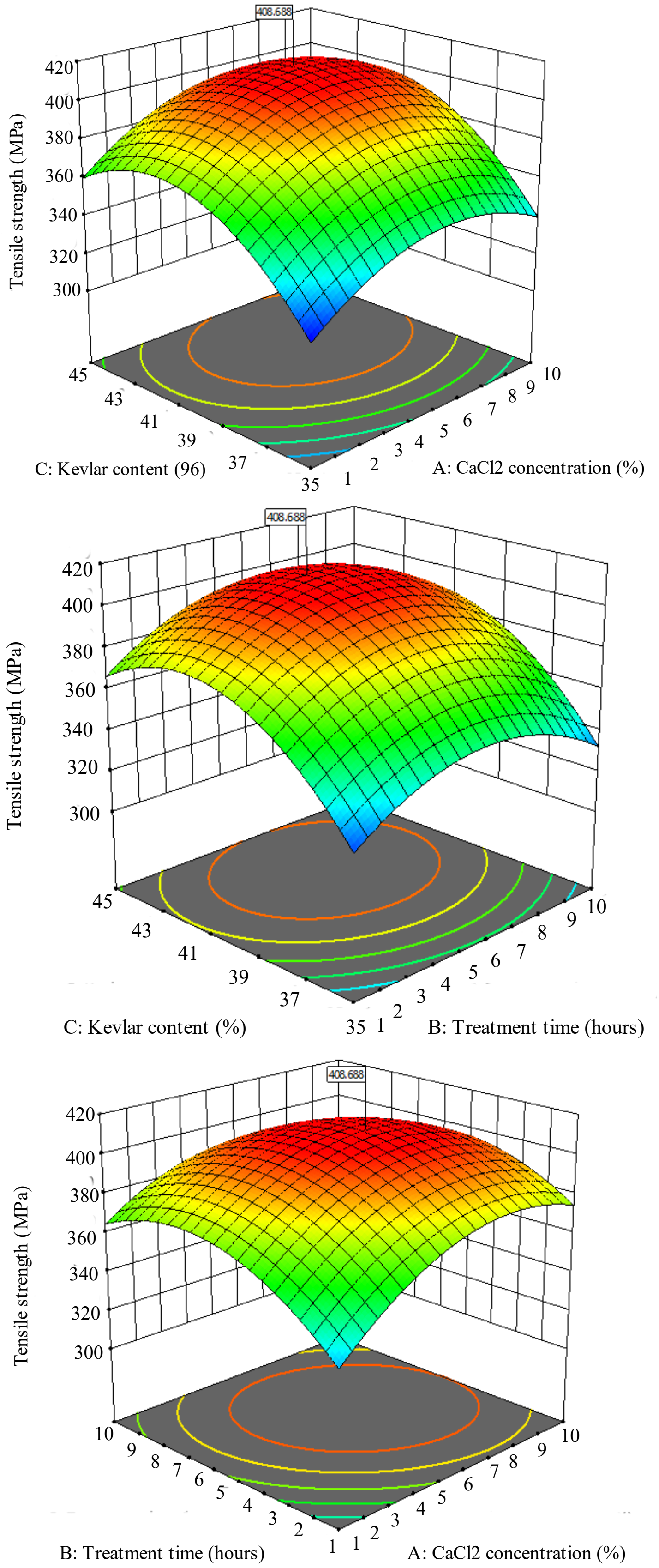

Figure 3. Response surface plot for the effects of variables on ts of the kevlar/epoxy polymer composites

The optimal preparation conditions extracted from Design Expert Software for the maximal TS were $CaCl_2$ concentration of 6.59 %, $CaCl_2$ treatment time of 5.58 h, Kevlar content of 42 %, and predicted TS of Kevlar/epoxy polymer composite was 408.7 MPa. The optimal treatment conditions of Kevlar fiber with $CaCl_2$ for the highest TS of Kevlar/epoxy polymer composites predicted from the model are in good accordance with the results reported in the literatures [23, 24, 31]. To confirm the validity of the suggested mathematical model, three parallel experiments were conducted under the predicted optimal conditions and showed an average value of tensile strength of 409.6 ± 3.8 MPa. This indicated that predicted and experimental data had a good fit and the model could be validated. The 3D response surfaces of the response using Eq. (3) are shown in Figure 3.

Structure and Interfacial Properties of Kevlar/Epoxy Polymer Composite

The structure and the interfacial properties of the sample obtained experimentally at predicted optimal conditions for the highest tensile strength ($CaCl_2$ concentration of 6.59 %, $CaCl_2$ treatment time of 5.58 h, Kevlar content of 42%) were investigated. Figure 4 shows the ATR-FTIR spectra of pristine Kevlar fiber and a Kevlar fiber treated with $CaCl_2$/EtOH solution.

The ATR-FTIR spectra of pristine Kevlar showed the peak at 3380 cm^{-1} which is assigned to N-H stretching vibration, the peak at 1640 cm^{-1} and 1515 cm^{-1} are stretching vibrations of C=O, the coupling peak of N-H bending vibration and C-N stretching vibration, respectively. The peak at 1308 cm^{-1} is attributed to the coupling between stretching C-N and bending C-H. The peak at 816 cm^{-1} is attributed to the absorption bands of the para-aromatic ring. The appearance of those peaks indicated the ontology structure of Kevlar fibers [23, 24]. Compared with the ATR-FTIR spectrum of pristine Kevlar, the ATR-FTIR spectra of treated Kevlar fiber are relatively similar. The complex formation between $CaCl_2$ and -C=O group was not observed at the ATR-FTIR spectrum of the treated Kevlar fiber as reported in reference [23]. The possible reason is the conditions for the treatment of Kevlar fiber are not strong enough to obtain freer N-H bonding which increased the intensity of the peak at 3380 cm^{-1} as reported in reference [23].

Figure 5 presents the XRD pattern of pristine Kevlar fiber and $CaCl_2$/EtOH-treated Kevlar fiber. The XRD pattern of pristine Kevlar fiber showed three peaks at $2\theta \approx 20.8^\circ, 23^\circ, 29^\circ$ corresponding to the (110), (200), and (211) planes, respectively [32]. After treatment with $CaCl_2$, no new peak appeared, indicating that the crystal type of Kevlar fibers did not change after treatment. The crystallinity degree X_{CR} of the treated Kevlar fiber was 77.3% lower than that of pristine Kevlar fibers of 80.2%. This is due to the damage of hydrogen bonding between –NH and -C=O groups. The results were in good accordance with the results reported in the literature [23, 24].

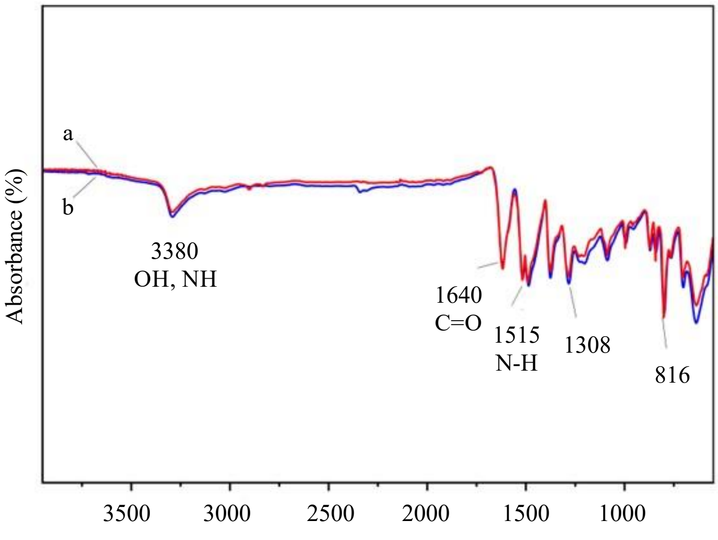

Figure 4. ATR-FTIR Spectra of (a) Pristine Kevlar Fiber, and (b) after Treatment with $CaCl_2$/EtOH.

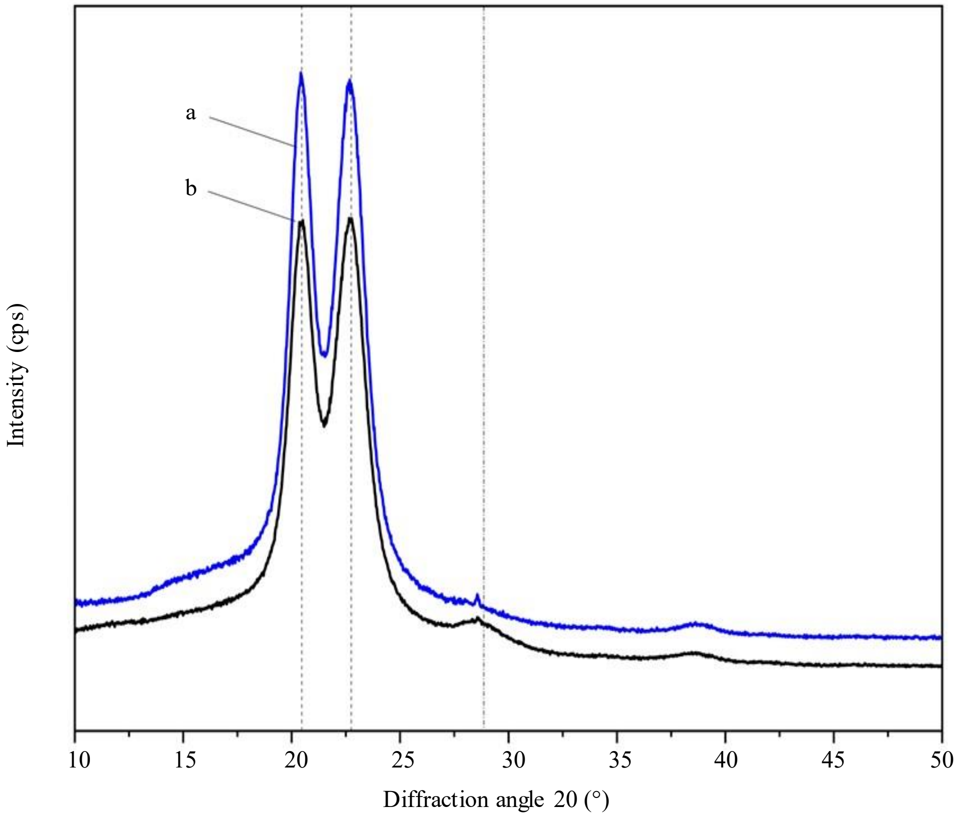

Figure 5. XRD Pattern of: (a) Pristine Kevlar Fiber and (b) after Treatment with $CaCl_2$/EtOH.

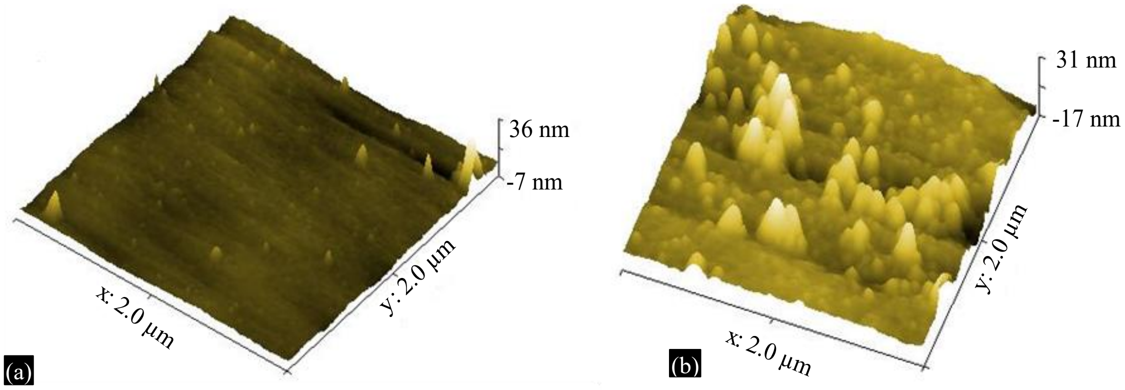

Figure 6. AFM Images of: (a) Pristine Kevlar Fiber and (b) after Treatment with $CaCl_2$/EtOH.

The AFM images (Figure 6) presented different surface morphology of the pristine Kevlar fiber and after treatment with $CaCl_2$/EtOH. While a relatively smooth surface of pristine Kevlar fiber is observed, a clear rougher surface of Kevlar fiber was recorded after the treatment with $CaCl_2$/EtOH solution.

The root mean square roughness (Rq) and arithmetic mean roughness (Ra) of pristine Kevlar fiber and after treatment with $CaCl_2$/EtOH were summarized in the Table 4.

Table 4. Roughness of pristine kevlar fiber and after treatment with $CaCl_2$

Number	Sample	Rq (nm)	Ra (nm)
1	Pristine Kevlar fiber	1,84	1,06
2	Treated Kevlar fibers	4,93	3,33

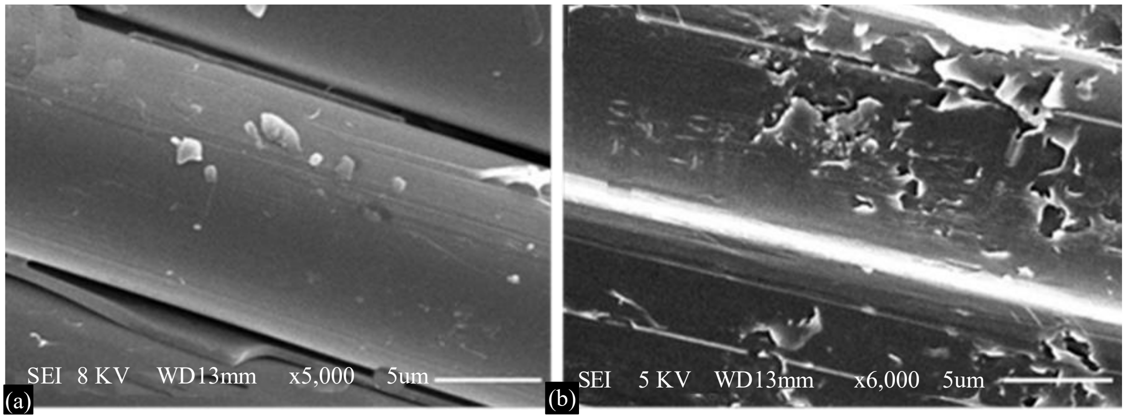

Figure 7. SEM images of (a) pristine kevlar/epoxy and (b) treated kevlar/epoxy composites.

The Rq and Ra of treated Kevlar fibers are significantly higher than that of pristine Kevlar fiber. The increased surface roughness of treated Kevlar fiber is attractive for reinforcing polymers, because the rougher surfaces would enable stronger mechanical interlocking (or anchoring) between the fibers and the matrix, and thus lead to improve the tensile strength of the polymer composite [13, 24].

The split tear strength (Ds) of Kevlar/epoxy polymer composite was employed to describe the interfacial bonding between the Kevlar fiber and the epoxy matrix. While the Ds value of pristine Kevlar/epoxy composite was 2.4 ± 0.1N/mm, Kevlar/epoxy polymer composites with treated fibers exhibited the split tear strength of 3.1 ±0.1 N/mm.

The improvement of the split tear strength of treated Kevlar/epoxy polymer composite was confirmed by SEM images of pulled out fiber (Fig. 7 a and b). Pristine Kevlar/epoxy polymer composite showed a smooth surface with a small amount of adhered epoxy resin. This result indicated that the interfacial bonding between Kevlar fiber and epoxy matrix was poor and the interface structure might not transfer stress satisfactorily [13, 21]. In contrast, the surface of the treated Kevlar/epoxy polymer composite showed some small globular-like microstructures, grooves, and the surface tiny tears along the longitudinal direction, resulting in strongly held by epoxy matrix. This could enhance the mechanical interlocking of the epoxy on the fibers' surface and improve the tensile strength of the polymer composites. Although the surface treatment of Kevlar fiber with $CaCl_2$/EtOH could enhance interfacial bonding between the Kevlar fiber and the epoxy matrix, no chemical bonds are formed at the interface of the polymer composites. Thus, the long-term performance of the polymer composites for different applications such as aerospace, automotive etc. maybe be affected.

CONCLUSIONS

Kevlar fiber was successfully modified with $CaCl_2$/EtOH solution to enhance interface interaction between the Kevlar fiber and the epoxy matrix. The response surface methodology using Box-Behnken Design was employed to predict optimal conditions for achieving the highest tensile strength of the high-performance Kevlar/epoxy polymer composite. The optimal parameters extracted by Design Expert Software were: $CaCl_2$ concentration of 6.59 %, $CaCl_2$ treatment time of 5.58 h, Kevlar content of 42 %, and the predicted tensile strength of 408.7 MPa. The tensile strength experimentally prepared at the optimal parameters was 409.6 MPa, indicating a good accordance between the predicted and actual experimental value. The crystallinity degree of the treated Kevlar fiber was slightly decreased compared to that of pristine Kevlar fiber. After being treated, the surface of Kevlar fiber became rougher. This increased roughness was the main reason for the significant improvement of the split tear strength from 2.4 N/mm of untreated Kevlar/epoxy polymer composite to 3.1 N/mm of the treated Kevlar/epoxy polymer composite. The improvement of interfacial bonding in the polymer composites is based on physical bonding. For long-term applications, both physical and chemical bonding at the interface of the polymer composites are desired and it needs to further investigate in the future.

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Author Contribution

Tran Thi Y Nhi: Conceptualization, investigation, writing-original draft; Trinh Duc Cong: investigation and methodology; Tran Thi Thanh Hop: investigation; Nguyen Thi Thuc: investigation; Dang Thi Mai: investigation; Tung Pham: conceptualization and editing; Ngo Trinh Tung: conceptualization, editing and supervision

Conflict of Interest

The authors declare that they have no conflicts of interest.

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