



# Optimizing Mechanical Performance in Woven Basalt Fibers/Epoxy Composites: Using Silane Coupling Agents to Modify Epoxy Resin for Fiber-Matrix Interface

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## Abstract

This study reveals that incorporating silane-coupling agents such as KH560, KH590, KH570, and KH550 into the resin matrix can improve the epoxy mechanical performance as well as the interfacial interaction between basalt fibers and modified epoxy. The increasing silane contents slightly increased thermal stability and char yields. The study also found that amino-silane coupling agents, particularly KH550, demonstrated better thermal stability enhancement. Pure epoxy had a T<sub>g</sub> of 91.4 °C, while epoxy had modified with 9wt% KH550 exhibited the highest T<sub>g</sub> of 98.7 °C. For instance, at 6 wt% concentration of KH560, KH590, KH570, and KH550, the flexural strength of the epoxy matrix increased by 69.4%, 57.6%, 83.4%, and 84.7%, respectively. When the content of KH590, KH570, and KH550 is 9 wt%, the tensile increases by 77.6, 49.0, and 79.6%, respectively. The W-BFs/epoxy composite flexural strength enhanced by 65.3, 81.5, and 74.5%, respectively. The compatibilizing agent would react with the silicon hydroxyl groups on the surface of the basalt fibers, as well as with the epoxide in the resin. This would create a Si-O-Si new bond, which would make the connection between the modified epoxy and the basalt fibers stronger. This process is favored for its cost-effectiveness and user-friendly operation.

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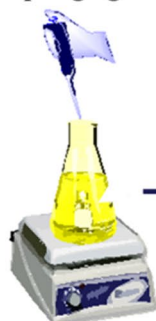
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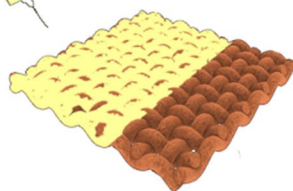
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## Graphical Abstract

Adding silane coupling agent



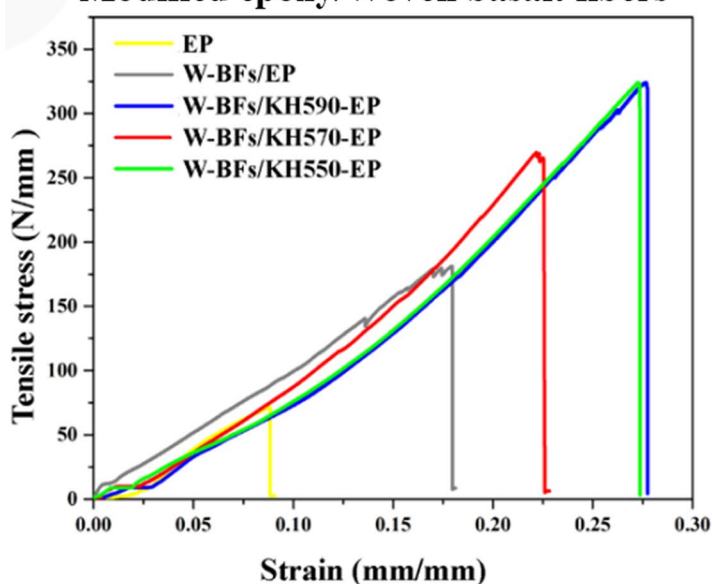
Mixing epoxy slolution



Woven basalt fiber/epoxy

Curing  
Cutting

## Modified epoxy/Woven basalt fibers



## Modified epoxy



**Keywords** Woven basalt fibers /epoxy composites · Cast-in-placed process · Silane coupling agents · Resin modification

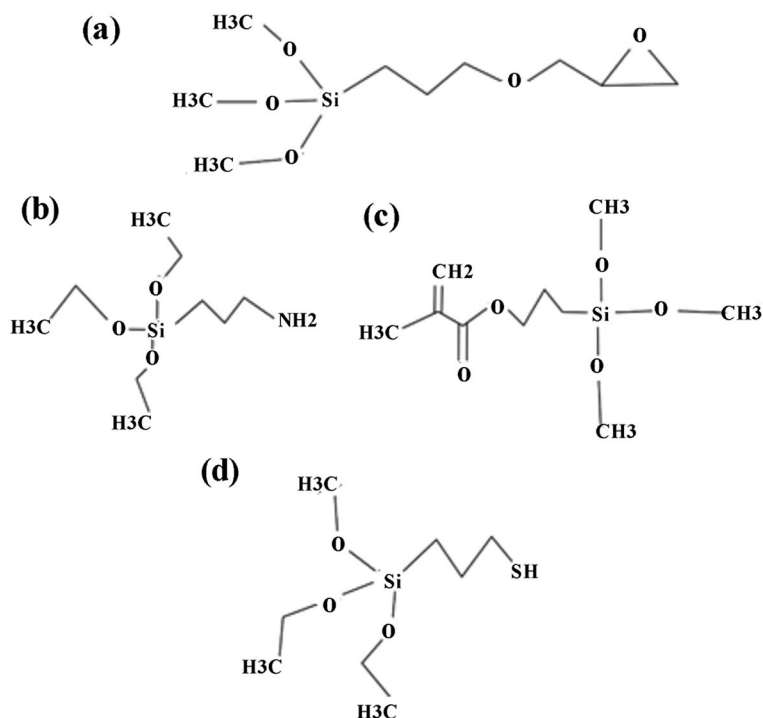
## 1 Introduction

Epoxy resins are ideal matrix systems for producing new composites of building materials and structural engineering because of their process ability, compatibility, and chemical resistance. However, the high cross-linking density of cured epoxy resins causes the material to become mechanically brittle due to a decrease in toughness, which ultimately contributes to the material's diminishing integrity and performance [1–7]. For high performance mechanical properties, the fibers need to be compatible with the matrix in a specific application [8–13]. The way polymer composites work depends on both the type of fillers and the degree of the nature of the interface. The nature of the interface

determines how well the load is transferred across the interface and how strong the functional fillers are in the polymer matrix. The robust fiber-polymer interface competently moves the external stresses [14, 15]. Therefore, active adhesion between the filler and the polymer has played a pivotal role in the performance of polymer-based composites and requires study to understand the performance of fillers in an epoxy matrix [16–18].

Consequently, researchers have conducted numerous studies aimed at enhancing the fiber/matrix interfacial bonding. Modification techniques could enhance the fiber/matrix bonding by altering the chemical composition of the filler surface [9, 19, 20]. Regrettably, chemical modification using chemical reagents could break down the

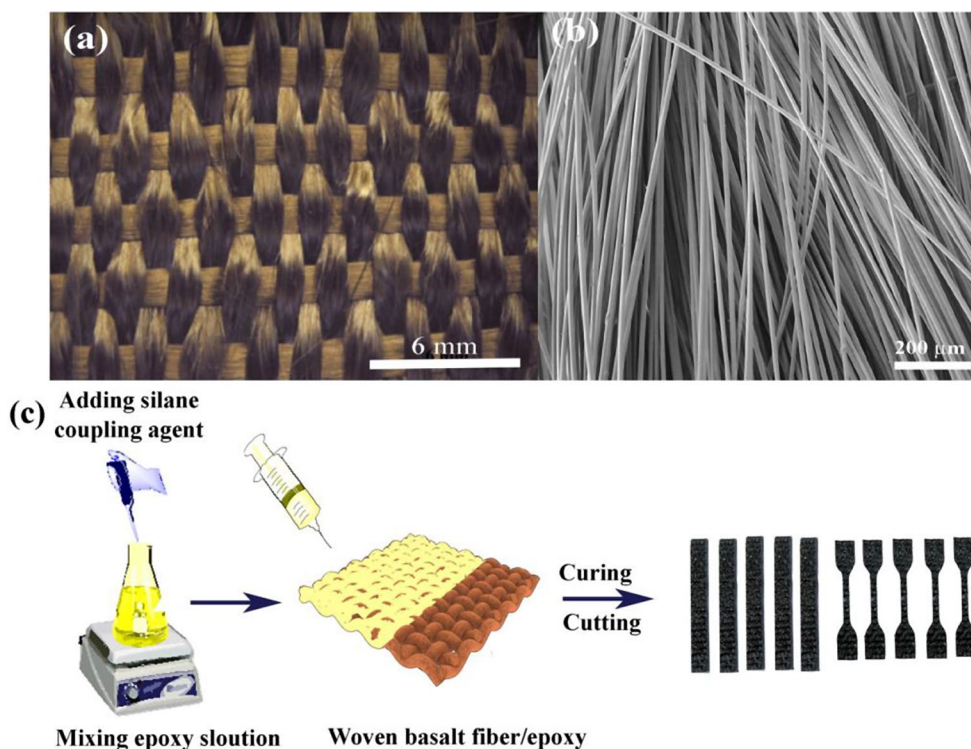
**Fig. 1** The chemical structures of silane coupling agents. (a) Glycidoxypropyltrimethoxysilane (KH560), (b)  $\gamma$ -Aminopropyltriethoxysilane (KH550), (c)  $\gamma$  methacryloyloxypropyltrimethoxysilane (KH570) and (d) 3-Mercaptopropyltrimethoxysilane (KH590)

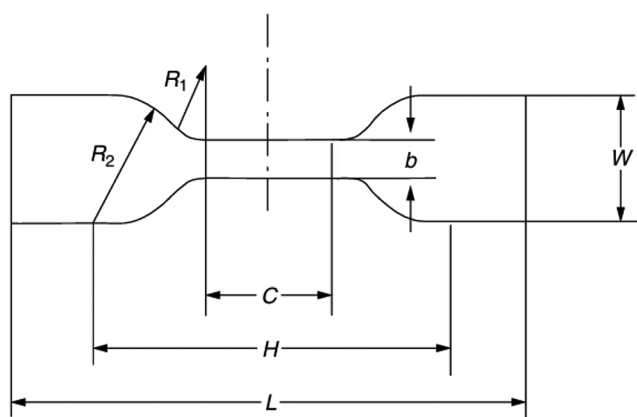


structure of the fibers, reducing their ability to strengthen the matrix [21]. To strengthen the polymer composite, researchers grew or stacked spherical, two-dimensional, or one-dimensional nanoparticles on the fiber surface. This method alters the topography of the fiber, increasing filler-specific surface area and matrix dispersion. Loading the nanofillers on the surface of the basalt fibers using a CVD

process reduces the mechanical performance of epoxy composites. This occurs because basalt fiber strength deteriorates when exposed to high temperatures during the CVD process. Exposure to high temperatures generally stimulates crystallization, resulting in a loss of mechanical properties for fibers. Besides, the operation condition is rigorous, not a green process, and the process involves costly production

**Fig. 2** Woven basalt fiber structure with cast-in-place epoxy composite production. (a) Woven basalt fiber optical photo, (b) SEM microscopy of the BFs and (c) Illustration depicting the steps involved in making epoxy composites with woven basalt fiber reinforcements





**Fig. 3** The shape and dimensions of the sample used for determining mechanical properties. The dimensions are as follows: length ( $L$ ) 115 mm, width ( $W$ ) 25 mm, ( $H$ ) 80 mm, width of the central section ( $b$ ) 6.5 mm, gauge length ( $C$ ) 33 mm, radius  $R_1$  14 mm, and radius  $R_2$  25 mm [31, 32]

and has not notably enhanced the mechanical performance of the composite [8, 22–24].

The agent of silane coupling has long been adopted with epoxy to strengthen the epoxy matrix and promote epoxy chain cross-linking. [25, 26]. Also, we believe that the silane coupling agents can increase interaction polymer chains with the basalt fiber network structure to enhance the bonding properties. Surface bonding increases when one molecule end binds to the reinforced surface and the function at the other side interacts with the phase of epoxy. Agents of silane coupling also can be reacted with the epoxy matrix [27]. Mixing epoxy with silane agents has two major benefits: (1) Silane coupling agents contain groups of alkoxy-silane that can adapt to the SiO-Si-based network structure of basalt fibers. A silane coupling agent is a silicate compound with a similar composition to the basalt fibers' surface. Silane may react with the silicon hydroxyl groups at the surface of basalt fibers, forming a new bond of Si-O-Si-. This bonding commonly affects the network structure of silicon oxide, leading to an increase in mechanical performance. Additionally, silane contains numerous functional groups that can cross-link with the epoxy matrix, thereby enhancing the fracture strength and flexibility of epoxy [28]. This characteristic ensures excellent agreement between the fibers surface and the epoxy matrix; (2) generally; silane agent is a solution with low-viscosity. As a result, the agent of silane coupling lowers the epoxy system viscosity with a high level of concentration as it gets incorporated into the resin and solidifies at room temperature, helping the epoxy penetrate quickly into the thicker forms of the fiber sheets [29].

The study's goal is to find out how different kinds and amounts of silane coupling agents, such as KH570, KH590, KH560, and KH550, affect the mechanical performance

of epoxy resin. The study also aims to understand how the molecular structure of the silane coupling agents influences the resin's toughness and how the degree of cross-linking, as indicated by changes in glass transition temperature ( $T_g$ ), correlates with the addition of different silane coupling agents. We used scanning electron microscopy (SEM) to evaluate the morphology of the epoxy resin before and after the addition of silane coupling agents, in order to understand changes in fracture behavior. Using silane coupling agents with different structures to modify epoxy resin improves interface, mechanical strength, and flexibility. These structures have distinct functional groups that interact specifically with the resin matrix, enhancing compatibility and obtaining specified features. Different silane structures allow for versatility in meeting various application needs and optimization through comparative testing, ensuring the modified epoxy resin meets durability and functionality criteria in specific contexts. Additionally, we cast the modified epoxy with silane agents within woven basalt fibers to enhance the interface between the matrix and fibers. Silane coupling agents create chemically appropriate reaction sites on fiber surfaces, strengthening the epoxy matrix. We use this procedure to prepare epoxy composites due to its low cost, good performance, and ease of usage.

## 2 Experimental Work

### 2.1 Materials

Commercial basalt-woven fabric from Ningbo Fangtai Glass Fiber Composite Material Co., Ltd. China having a diameter of 13.0  $\mu\text{m}$  and thickness of 3 mm was used. The SEM and macroscopic images of the woven basalt fibers are shown in Fig. 2a and b.

The epoxy resin (EPON 862) and (DETDA) were imported from Dongguan Qiancheng plasticizing materials Co., Ltd, China. EPON 862 is a low viscosity (1.2  $\text{g}/\text{cm}^3$ ) [30].

(3-Glycidoxypropyl)trimethoxysilane (KH560), 3-mercaptopropyltriethoxysilane (KH590),  $\gamma$  methacryloyloxypropyltrimethoxysilane (KH570), and  $\gamma$ -aminopropyltriethoxysilane (KH550) were supplied by Chengdu Kelong Chemical Reagent Factory, Chengdu, China. All the reagents were used without further purification. Chemical structure of the silane coupling agents is shown in Fig. 1.

### 2.2 Epoxy Resin Modification by Silane-Coupling Agent

Enhanced cross-linking of the epoxy matrix molecules was achieved by combining epoxy resin with different structures

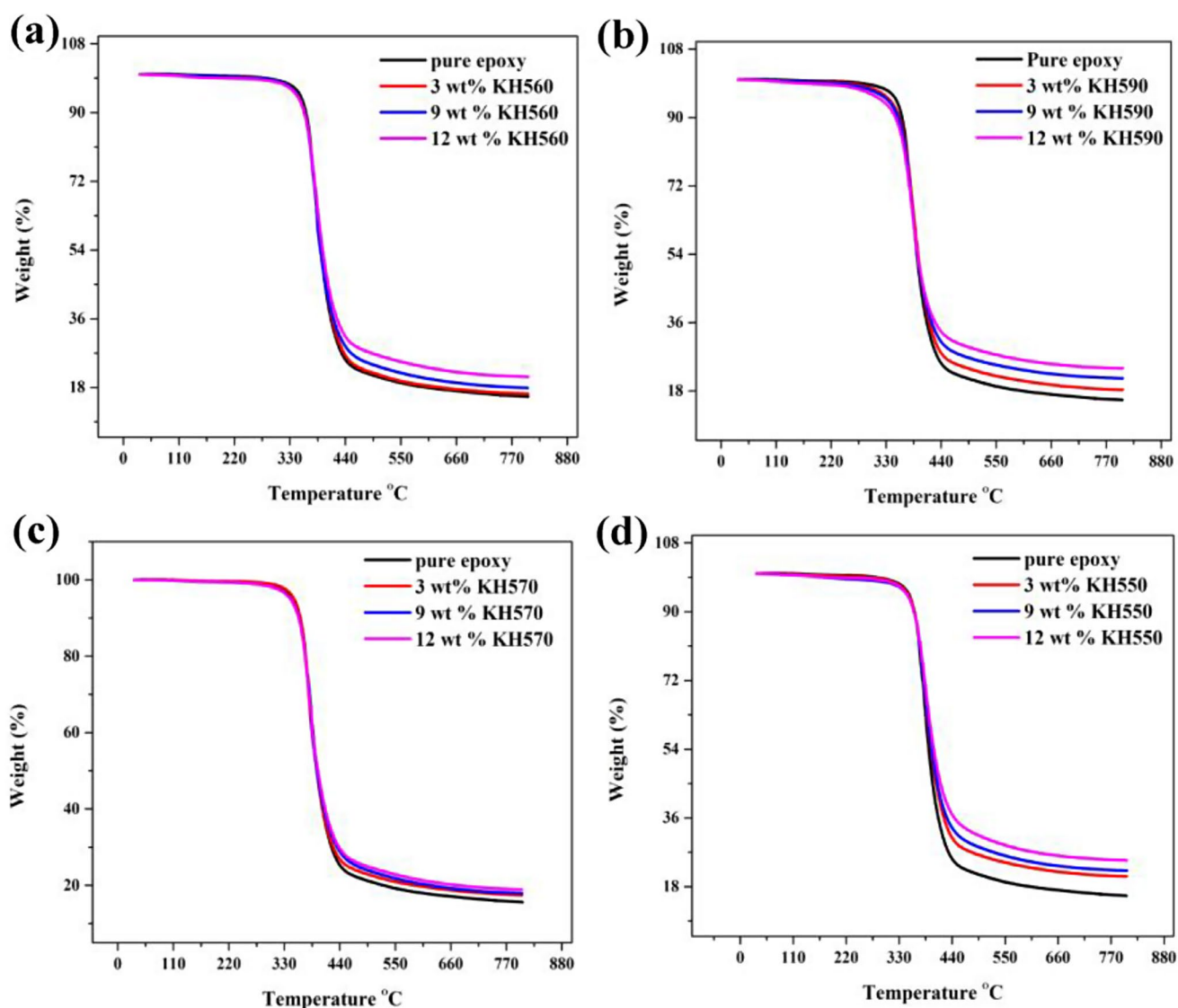


## 2.4 Characterization and Testing

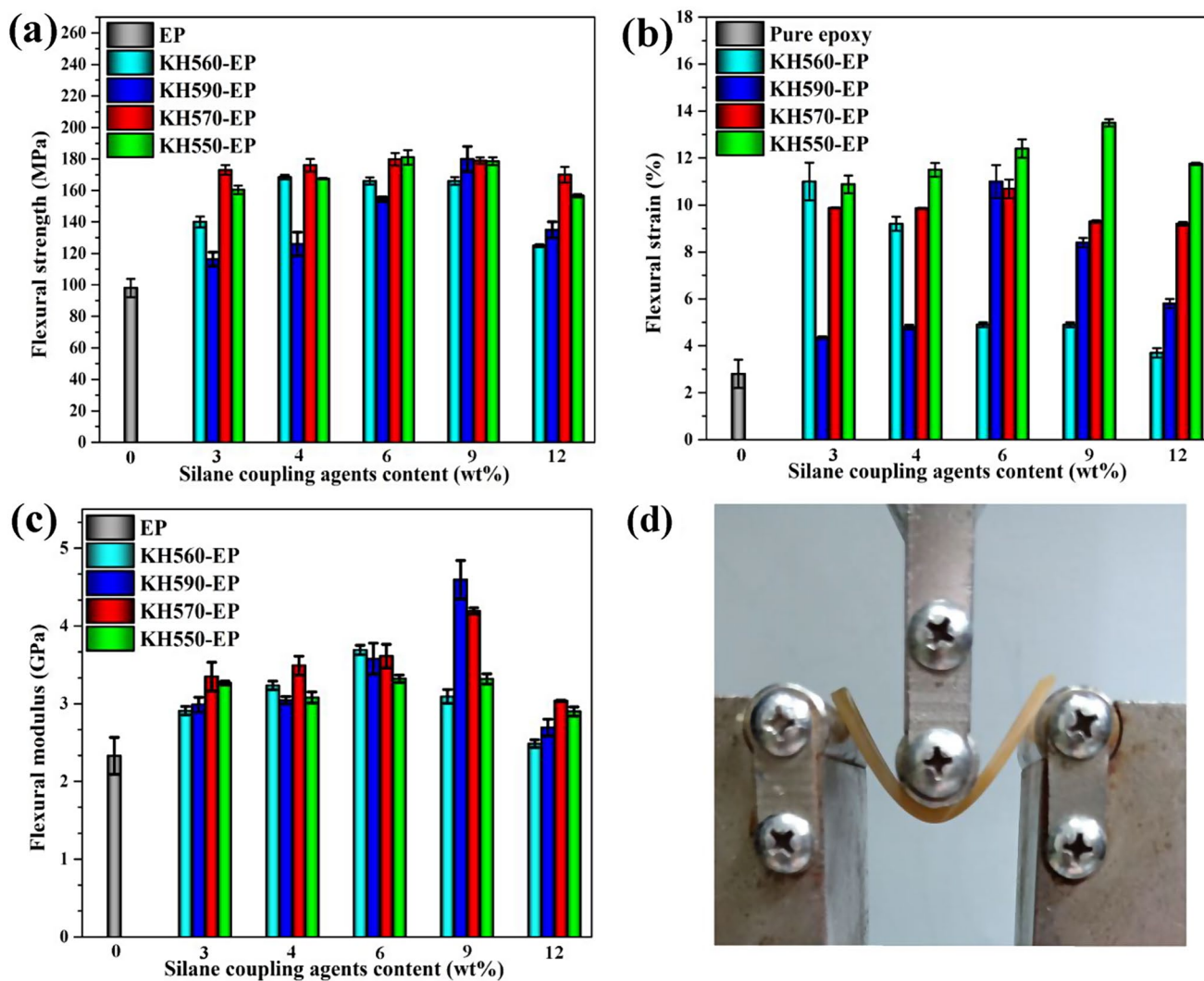
Using a FE-SEM, model JEOL JSM-7001 F (made in USA), Basalt fibers and their embedded resin composites were analyzed for morphology and fracture surface analysis. The FTIR Nicolet 5700 (made in USA) was used to evaluate the chemical composition of both virgin basalt fibers (BF) and silane-functionalized epoxy/BF epoxy composites.

Thermal gravimetric analysis (TGA) was utilized to measure the temperature of decomposition of the filler and modified resin. TGA was done using a thermo-analysis technique (STA449F3, NETZSCH, Selb, Germany). Using nitrogen, the temperature rose 10 °C/min from ambient to 800 °C.

A CMT4304 machine from SUST, Sansitaijie, Guangdong, China, and a universal testing equipment (PWS 100) made in China were used to assess the flexural and tensile properties, respectively. Following the guidelines laid out in GB1040-92 and GB1449-2005, The sample for the flexural test has dimensions of 4 mm in thickness, 10 mm in width, and 80 mm in length. The sample for the flexural test has dimension shown in Fig. 3, the cross head speed was set at 2 mm/min. All the presented results are the average value of five specimens.



**Fig. 5** Thermogravimetric analysis curves of pure epoxy and the silanized epoxy resin. (a) EP-KH560, (b) EP-KH590, (c) EP-KH570 and (d) EP-KH550



**Fig. 6** Flexural properties of epoxy (EP) modified with different concentration of silane coupling agents, 3-Glycidoxypropyltrimethoxysilane Silane (KH560), 3-Mercaptopropyltriethoxysilane (KH590), 3-Methacryloxypropyltrimethoxysilane (KH570) and 3-Aminopropyltriethoxysilane (KH550).

## 3 Results and Discussions

### 3.1 Chemical Structure of Silanized Epoxy Resin

The reason for mixing the epoxy resin with various structures of silane coupling agents is to improve the cross-link among epoxy chains by the formation of Si-O-Si bonds. FTIR spectra can be used to define the functional group and demonstrate different vibrational patterns of different bonds linked with chains from epoxy matrix and basalt fibers. The typical FTIR bands for pure epoxy resin are shown in Fig. 4a-d. The peaks at 1508 and 1609  $\text{cm}^{-1}$  are refer to C-C and C=C, respectively. The peak in accordance with stretching of epoxide ring C-H is at 2924/2354  $\text{cm}^{-1}$ . The peaks at 1021  $\text{cm}^{-1}$  are designated to the epoxide group

(C-O-C). As shown in Fig. 4b-d, it was also evident that the peaks intensity of -OH at 3430–3742  $\text{cm}^{-1}$  increased after mixing epoxy resin with silane coupling agent as a result of the creation of new groups of hydroxyl after the open-ring. The epoxide group reaction confirmed the interaction between the phalanx coupling agents and the epoxy. From Fig. 4a, the FTIR spectra of 3-glycidoxypropyltrimethoxysilane (KH560)-modified epoxy show new peaks. The absorption peaks at 3742  $\text{cm}^{-1}$  refer to the hydroxyl group. Also, new peaks actively verify that the KH560 coupling agent reacted with epoxy appearing at 1273, 1107 and 1027  $\text{cm}^{-1}$ , which are designated to the Si-C, Si-O-C and Si-O-Si bands, respectively.

The spectra of 3-Mercaptopropyltriethoxysilane (KH590) modified epoxy is displayed in Fig. 4b. After modification epoxy by KH590, some new peaks appear including Si-O-Si

(C-O-C). As shown in Fig. 4b-d, it was also evident that the peaks intensity of -OH at 3430–3742  $\text{cm}^{-1}$  increased after mixing epoxy resin with silane coupling agent as a result of the creation of new groups of hydroxyl after the open-ring. The epoxide group reaction confirmed the interaction between the phalanx coupling agents and the epoxy. From Fig. 4a, the FTIR spectra of 3-glycidoxypropyltrimethoxysilane (KH560)-modified epoxy show new peaks. The absorption peaks at 3742  $\text{cm}^{-1}$  refer to the hydroxyl group. Also, new peaks actively verify that the KH560 coupling agent reacted with epoxy appearing at 1273, 1107 and 1027  $\text{cm}^{-1}$ , which are designated to the Si-C, Si-O-C and Si-O-Si bands, respectively.

and Si-C stretches at 1106 and 1171  $\text{cm}^{-1}$ , respectively. The FTIR spectra of 3-Methacryloxypropyltrimethoxysilane (KH570) modified epoxy were also shown in Fig. 6c. The appearance of the peak at 815, 1032, 1295, 1110, 1714  $\text{cm}^{-1}$  in proportion to Si-O, Si-O-Si, Si-O-C and C=O, respectively. Also, Fig. 4d gives the KH550 spectra provided by the FTIR, epoxy after and before modification. The spectrum of 3-Aminopropyltriethoxysilane (KH550) is displayed several peaks containing N-H stretches at 1575 and 3421  $\text{cm}^{-1}$ , C-N stretches at 1167  $\text{cm}^{-1}$ , Si-O-C stretches at 1080  $\text{cm}^{-1}$ , and Si-O stretches at 950  $\text{cm}^{-1}$ , the stretch absorptions bands of N-H at 3421  $\text{cm}^{-1}$ . The existence of N-H at 1637  $\text{cm}^{-1}$  is mostly correlated to the amine group introduced by KH550. Besides, the absorption peak at 1173, 1038, 1110 and 1815  $\text{cm}^{-1}$  belong to the C-N, Si-O-Si and Si-O groups, respectively, which exhibits the 3-Aminopropyltriethoxysilane (KH550), is chemically cross-linked with epoxy. It can be observed that the network was entirely cured as judged by the demise of the epoxide peak and the show of OH peak. However, adding different silane coupling agents to epoxy resin causes changes that can be seen and the appearance of new peaks in the FTIR spectrum compared to the peaks for pure epoxy. This shows that there are important chemical interactions and changes happening within the composite material. These changes and new peaks in the FTIR spectrum show that adding certain silane coupling agents to the epoxy resin changed the material and made it easier for networks to connect.

### 3.2 Thermal Degradation of Silanized Epoxy Resin

Thermogravimetric investigation is commonly used to examine the thermal stability of polymer materials. Through strong chemical connections in the polymer matrix, silane

agents boost cross-linking density and structural integrity in epoxy resins, improving thermal stability. Due to its heat tolerance, silicon in silane compounds inhibits oxidative degradation and reduces weight loss at high temperatures. If decomposed, silane-modified epoxies create more char, which protects against heat deterioration. High temperatures produce and break hydrogen bonds, causing controlled breakdown. Silane agents improve epoxy resin thermal resilience, making them appropriate for high-temperature applications. Figure 2 and Table 1 display the results of the TGA analysis of the cured samples. It was clearly observed that the thermal stability of the cured resin rose slightly with the rise in silane contents in the epoxy. As the silane coupling agent contents rise from 0.0 to 12.0 wt%, the onset decomposition temperature increases from 356.0 °C for pure epoxy to approximately 357.0–360.5 °C for the modified epoxy. Moreover, the char yields of the epoxy also rise with the increase in silane coupling agents. The modified epoxy increased the char yields from 15.1% for pure epoxy to approximately 16.3–24.8%. There were more chemical bonds in the epoxy as the silane group content went up, which was proportional to the silane coupling agent content. The high heat resistance of the silicon element, where silica significantly inhibits the weight loss of oxidative polymers and results in a higher residual weight of the polymer at 800 °C with increasing silane coupling agent contents, could have contributed to the improved thermal stability. Also, Table 1 shows that the temperature decomposition of epoxy decreased slightly with an increase in silane coupling agent epoxy to 12 wt% compared with 3 wt%. This is because silanol groups have a tendency to form hydrogen bonds with each other, and the decomposition temperature will increase as the temperatures rise above 300 °C during the thermal degradation test. With rising temperatures, epoxy resin's chemical composition changes, and molecular movement will accelerate, increasing the distance between molecules. The disruption of hydrogen bond production led to a significant decrease in the number of hydrogen bonds [21]. Therefore, the reduction of hydrogen bonds will inescapably cause a decrease in the decomposition of modified epoxy. The data also showed that amino-silane coupling agents (KH550) can better make the epoxy resin bond more thermally stable than epoxy that has been improved with KH560, KH570, or KH90. One could consider KH550 as a component of the toughened that cross-links with the epoxy [22]. KH550 reacts with epoxy, which might create a more chemical bonding structure in contrast to KH560, K570, and KH590. It could be a cause for the best modifying influence of KH550 on the flexural performance of the resin modified by silane coupling agents [23].

**Table 1** TGA and tg analysis data of the cured pure and modified epoxy resin

Samples	Onset Decomposition Temperature (°C)	Char yields at 800 °C (%)	Glass transition temperature (T <sub>g</sub> , °C)
Pure epoxy	356.0	15.1	91.4
3wt% KH560	360.0	16.3	81.1
9 wt% KH560	359.6	17.8	66.5
12 wt% KH560	359.2	20.7	61.8
3 wt% KH590	356.4	18.1	67.7
9 wt% KH590	355.5	21.2	66.8
12 wt% KH590	351.6	24.0	64.0
3 wt% KH570	358.9	17.4	92.3
9 wt% KH570	359.0	17.9	90.5
12 wt% KH570	356.1	18.9	83.7
3 wt% KH550	360.0	20.7	88.1
9 wt% KH550	360.5	22.6	98.7
12 wt%KH550	357.0	24.8	97.5

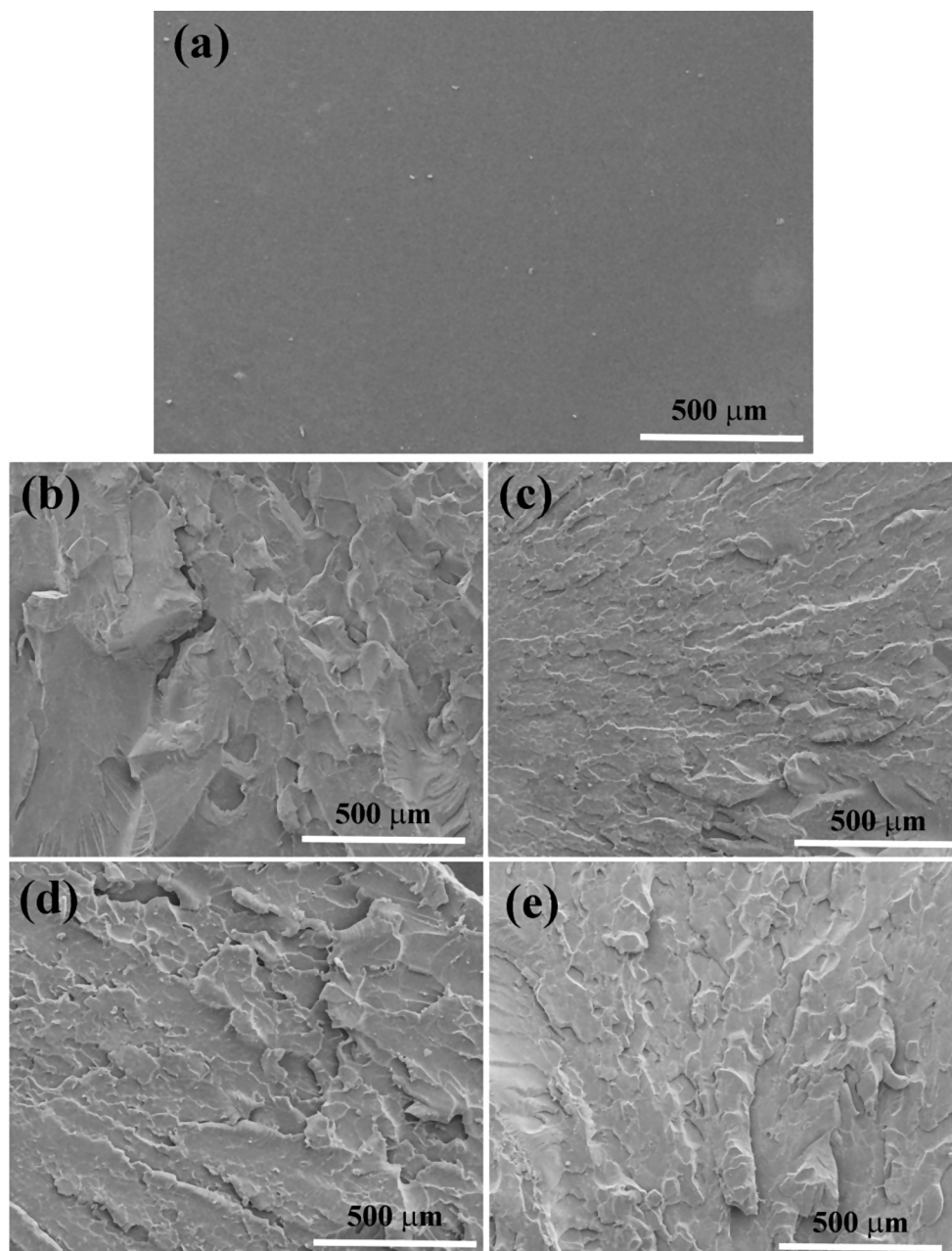


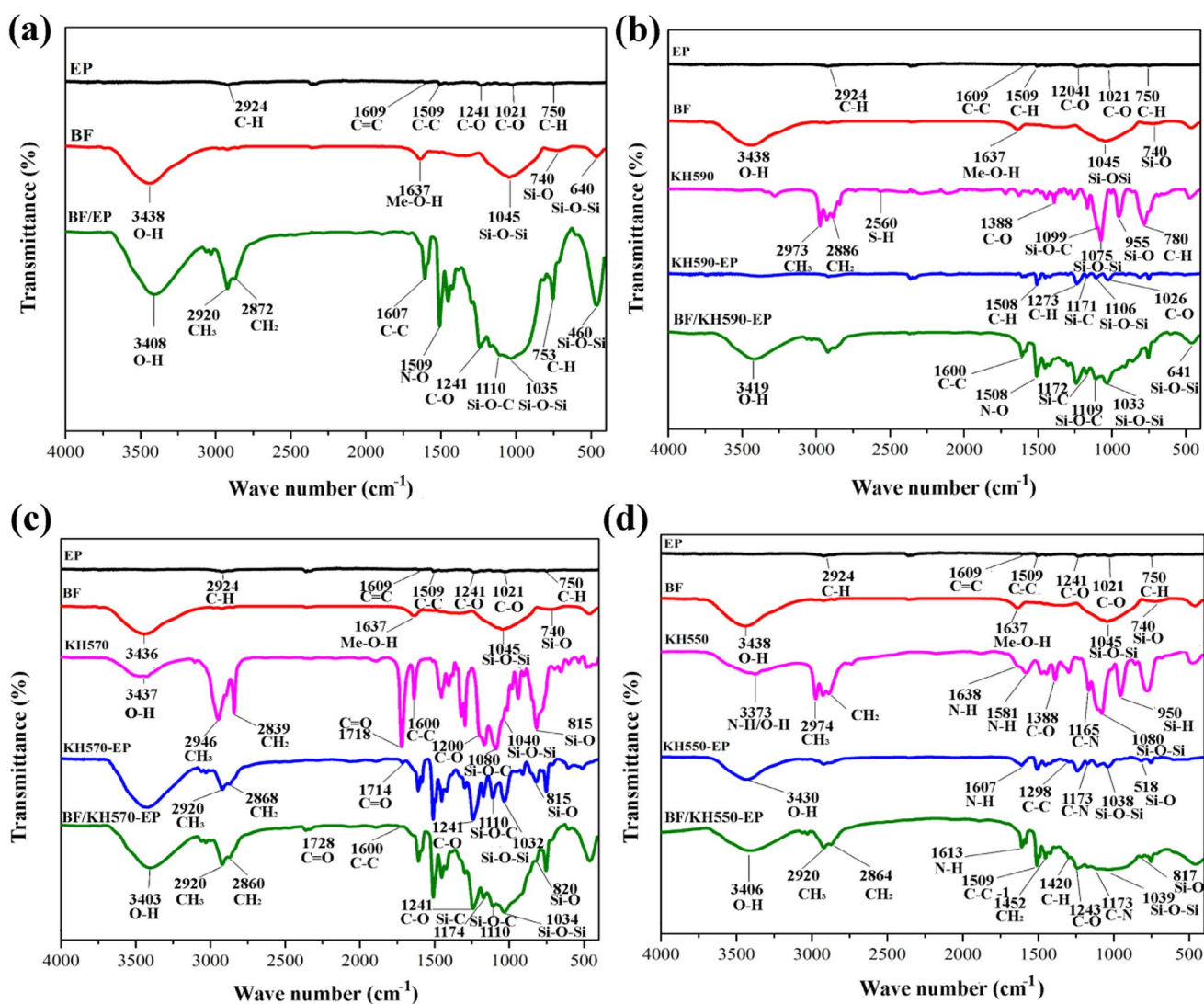
### 3.3 Dynamic Mechanical Analyses of the Silanized Epoxy Resin

Glass transition temperature ( $T_g$ ) is a basic guideline for polymers, clearly related to the degree of cross-linkage: when cross-linking is higher,  $T_g$  is higher [33]. The  $T_g$  data for the different structures and concentrations of silane coupling agents mixed with epoxy are displayed in Table 1. The  $T_g$  of the cured pure epoxy was 91.4°C. The epoxy resin modified with 9wt% KH550 showed the highest value of glass transition temperature, reaching 98.7°C. As the concentration of imide groups increases, the stiffness of the epoxy chains also increases, leading

to a higher rotational barrier. This ultimately results in an enhancement of the glass transition temperature of the epoxy [34]. Table 1 illustrates that the  $T_g$  values of epoxy modified with KH590, KH570, and KH560 drop from 91.4 °C (pure epoxy) to approximately 64.0–90.5 °C. When there was more than a certain amount of flexible silane monomer in the resin structure, it made the epoxy chain more mobile by replacing some rigid phenyl structures with flexible silane molecules. This made the treated material less flexible [35, 36].

**Fig. 7** SEM images of epoxy fracture surfaces (EP). (a) EP, (b) EP-KH560, (c) EP-KH590, (d) EP-KH 570 and (e) EP-KH550





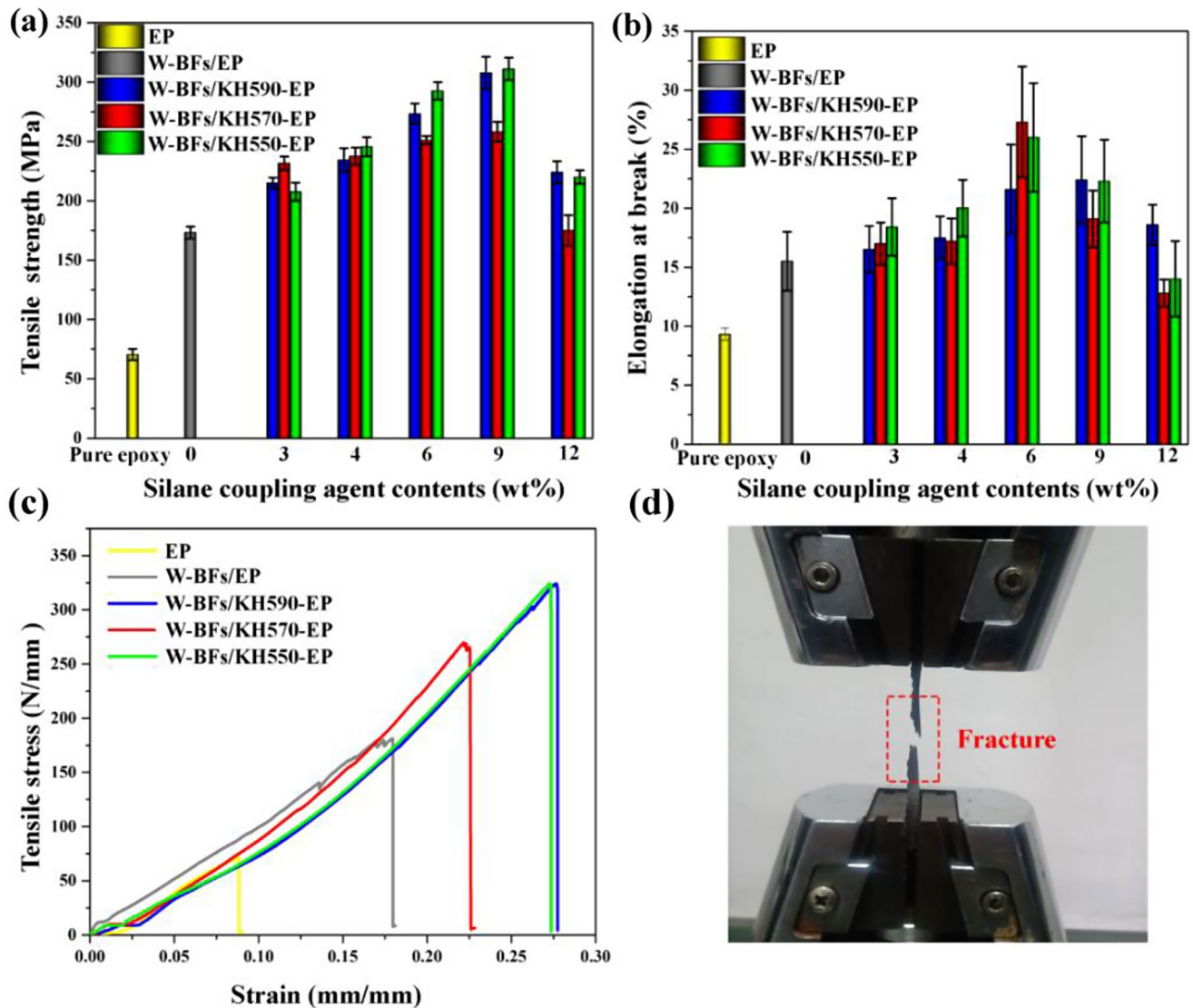
**Fig. 8** FTIR spectra of the pure epoxy (EP) and silane coupling agents/epoxy-basalt fiber. (a) Epoxy matrix modified by basalt fiber (BF/EP), (c) epoxy matrix modified by 3-Mercaptopropyltrimethoxysilane and basalt fiber (BF/KH590-EP) and basalt fiber, and (d) epoxy matrix

modified by 3- Methacryloxypropyltrimethoxysilane and basalt fiber (BF/KH570-EP) and (e) epoxy matrix modified by 3-Aminopropyltriethoxysilane and basalt fiber (BF/KH550-EP)

### 3.4 Mechanical Performance of Silanized Epoxy Resin

The mechanical performance has been studied to observe the effect of epoxy mixed with various silane coupling agent concentration and types on the mechanical characteristics. Experimental data of flexural strength, flexural modulus and strain at break are displayed in Fig. 6. Figure 6a shows an improvement in the composite flexural strength as the loading of agents raised. The epoxy flexural strength blended with 6 wt,% of KH560, KH590, KH570 and KH550 was strengthened by 69.4%, 57.6%, 83.4% and 84.7%, respectively. When silane coupling agents were combined with epoxy resin, they were able to interact with the resin by bonding with the epoxide groups and silanol groups. This

allowed them to become part of the cross-linked network, serving as crosslink sites to a certain degree. As a result, this led to improved cross-linking and therefore increased flexural strength. Whereas, the flexural strain improved by 75.0, 292.8, 281.8 and 342.8% with the same silane coupling agent concentration (6wt%), respectively. The flexural strain of the samples is shown in Fig. 6b and d. When the silane coupling contents were raised, the strain of epoxy significantly increased. It is assigned to the fact that the silane monomers improve the cross-linking between the epoxy chains through chemical bonding. However, increasing the number of polysiloxane (Si-O-Si) segments in the epoxy's network demonstrates the excellent elasticity of the polysiloxane [37]. As shown in Fig. 6c, flexural modulus was increased with the silane coupling agent content. From



**Fig. 9** Measured the tensile characteristics of the as-prepared woven basalt fiber/epoxy composites before and after the adding silane coupling agents (KH590, KH570 and KH550) in epoxy resin. **(a)** strength,

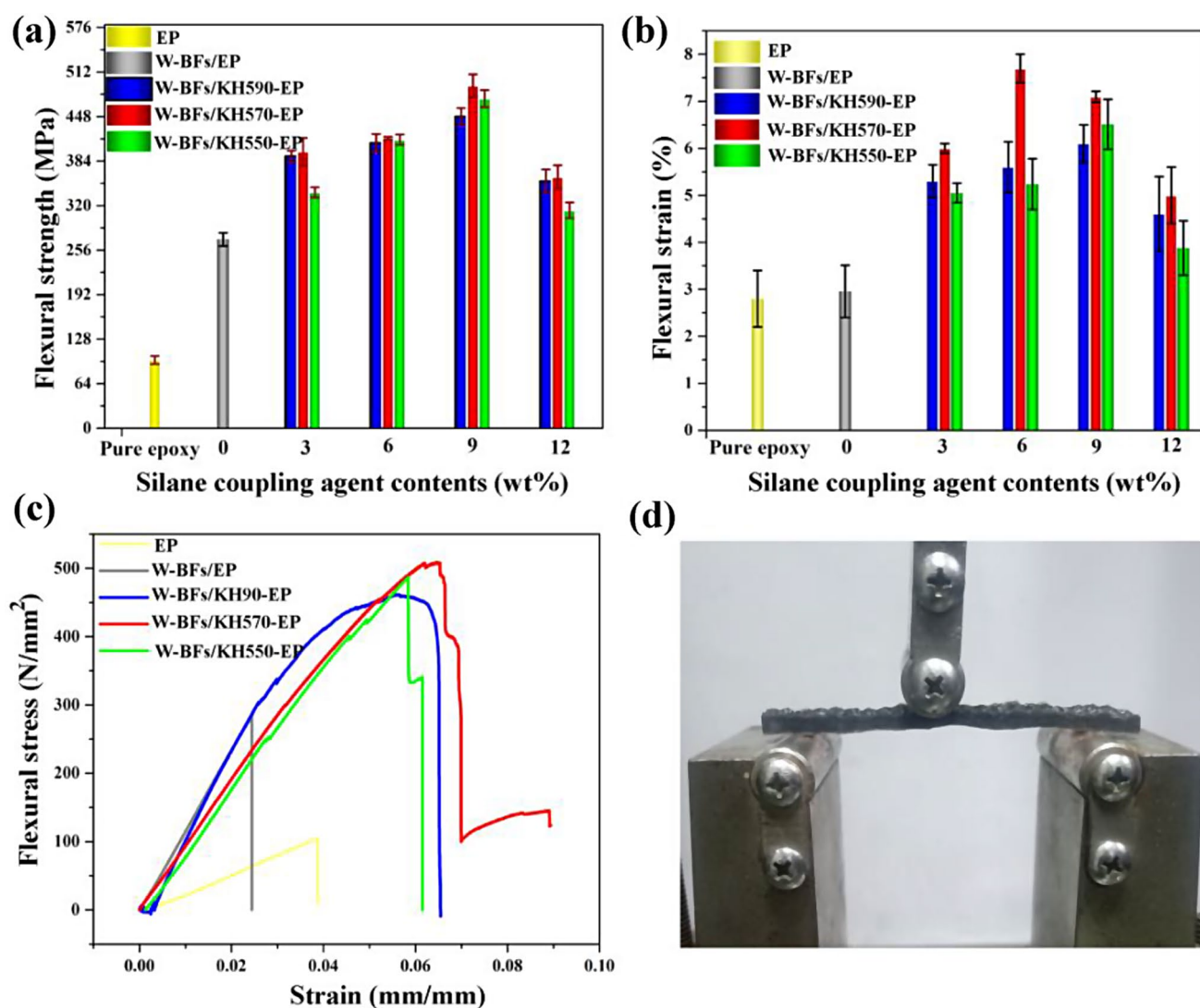
**(b)** strain and **(c)** stress-strain curve of epoxy/composite and **(d)** tensile testing on a woven basalt fibers/epoxy composite

Fig. 6c, it can be concluded that flexural performance was mostly higher when the epoxy contained 9wt% KH790. When the content of the silane coupling agent is more than 9wt%, it leads to a decrease of the flexural properties. This is due to the high amount of silane coupling agent, which may establish a weak cross-link by physical adsorption. The surplus silane layer has the potential to address imperfections in the composite interphase. As a result, the flexural characteristics of the composites deteriorate as the concentration of silane coupling agent increases [36, 38]. These findings suggest that all four silane coupling agents, KH570, KH550, KH560, and KH590, played vital roles in improving the mechanical properties of epoxy resin composites. However, KH570 and KH550 demonstrated superior performance compared to KH560 and KH590, particularly

in enhancing flexural strength and strain at break. These results highlight the potential of KH570 and KH550, along with the other silane agents, for significantly improving the mechanical performance of epoxy resin composites.

### 3.5 Strengthening Mechanism of the Silanized Epoxy Resin

The morphology of the pure and silanized epoxy by 9wt% of coupling agents (KH560, KH590, KH570 and KH550) was confirmed using SEM examination Fig. 7. When the epoxy was directly mixed with silane coupling agent liquids, the silane molecules were homogeneously dissolved within the epoxy, due to the excellent compatibility of the silane coupling agent with epoxy matrix. As shown in Fig. 7a, the



**Fig. 10** Measured the flexural performance of the woven BFs/epoxy composites before and after the added silane coupling agents (KH590, KH570 and KH550) in epoxy resin. (a) strength, (b) strain, (c) stress-strain curve, and (d) three-point bending test of woven basalt fibers/epoxy composite

fracture surface of pure epoxy is comparatively glossy, this is a representative fracture manner for the brittle thermosetting polymer. Aside from that, the cracks spread freely, exposing their weak nature to crack formation and propagation. Accordingly, the fracture process of pure epoxy exhibited brittle fracture behavior.

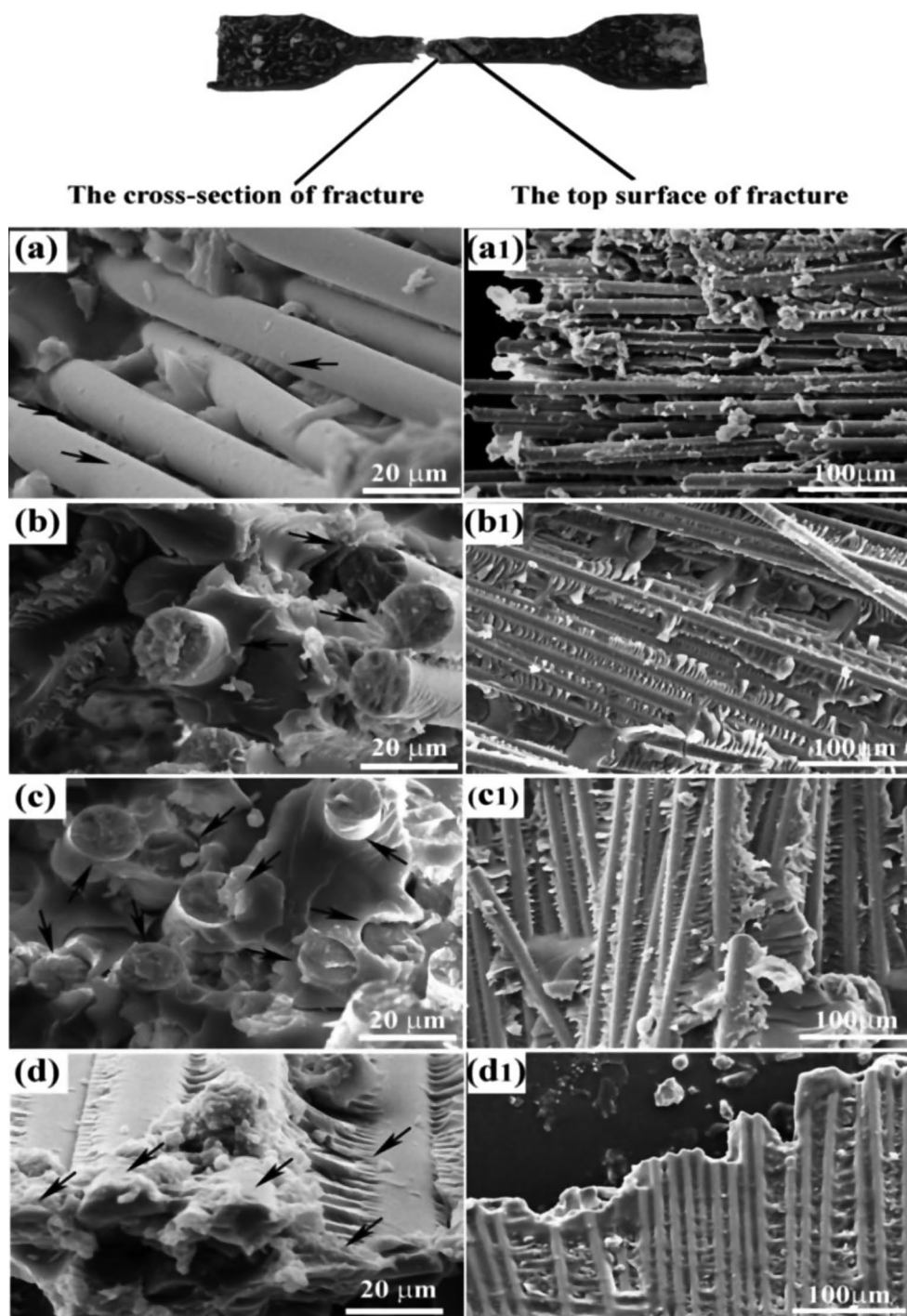
On the contrary, the epoxy modified with silane coupling agent exhibits a rougher fracture surface, and numerous tortuous and indentations can be observed (Fig. 7b-e), referring to flexible fracture behavior. The silane groups significantly improve the flexibility among epoxy chains, leading to plastic deformation when exposed to external stresses. When the silane coupling agents are mixed with the epoxy resin, they can interact with epoxy resin and curing agent by their C-O-C and Si-OH and join into the system through chemical

bonds, which can act as cross-link locations in some locations and cause an increased density of cross-link.

### 3.6 Chemical Structure of Silanized Epoxy/Woven Basalt Fibers Composites

Figure 8 shows the FTIR spectra for the epoxy (EP), as received basalt fibers (BF), modified epoxy matrix by three kinds of silane coupling agents (KH590-EP, KH570-EP and KH550-EP) separately, and the epoxy composite hybrid with silane coupling agents and basalt fiber (BF/KH590-EP, BF/KH570-EP and BF/KH550-EP). The FTIR spectra of basalt fiber (BF) are shown in Fig. 8a-d. A group of high intensive peak is shown in the zone of  $1045\text{ cm}^{-1}$ . The bands can be assigned to the Si-O-Si network vibrations. In the range of  $1637\text{ cm}^{-1}$  a peaks with medium intensities can be

**Fig. 11** SEM images of fracture surface at low and high magnification. **(a** and **a1)** Epoxy composite modified by as received woven basalt fibers (W-BFs/EP), **(b** and **b1)** epoxy composite modified by 3-mercaptopropyltriethoxysilane and woven basalt fibers W-BFs/KH90-EP, **(c** and **c1)** epoxy composite modified by 3-methacryloxypropyltriethoxysilane and woven basalt fibers W-BFs/KH570-EP and **(d-d1)** epoxy composite modified by 3-Aminopropyltriethoxysilane and woven basalt fibers (W-BFs/KH550-EP)



assigned to the Me-O-H bonds, herein, Me stands for metals which are basalt fiber constituents. Also, it was observed that in the case of (BF/EP, BF/KH590-EP, BF/KH570-EP and BF/KH550-EP), the band intensity at  $3403\text{--}3419\text{ cm}^{-1}$  related to O-H group improved in comparison with that of pure epoxy (EP). This demonstrates the epoxide reaction via formation of hydroxyl groups with the basalt fiber and silane agents, as shown in Fig. 8a-d. The reduction in the absorption peak intensity of the Si-O-Si ( $1034\text{--}1039\text{ cm}^{-1}$ )

and Si-O-C groups ( $1109\text{--}1110\text{ cm}^{-1}$ ) in comparison to the absorption peak intensity of the silane coupling agent and the basalt fiber verified the activity among the epoxy, silane coupling agent, and basalt fiber. If the epoxy and agent of silane coupling react with the basalt fibers' silicon hydroxyl groups and the resin's epoxide, they can form a new bond of -Si-O-Si-, which strengthens the link between the basalt fibers and epoxy.

### 3.7 Mechanical Properties of Silanized Epoxy/Woven Basalt Fibers Composites

Figure 9a-d shows the strength of the tensile, strain and stress-strain curve of pure epoxy and W-BFs/EP composites. The unmodified W-BFs/EP composite is higher 146.0% than neat epoxy. As displayed in Fig. 9a, the infusing of silane coupling agent and epoxy into woven basalt fibers has a remarkable effect on the tensile performance of W-BFs/EP composites. The addition of KH590, KH570 and KH550 remarkably raises the composites' tensile strength, in comparison with the pure EP/BFs composite. Compared with pure W-BFs/EP composite (173.2 MPa), when the content of KH590, KH570 and KH550 is 9wt%, the tensile strength reaches a maximum 307.6, 258.1 and 311.0 MPa and increases by 77.6%, 49.0% and 79.6%, respectively. Besides, the tensile strength and strain increases according to the silane coupling agent concentration inside the epoxy. Figure 9b explains that the tensile strain of neat epoxy, pure W-BFs/EP and silanized W-BFs/EP composites versus the silane coupling agent contents. Sulfuric acid coupling agents significantly increase the tensile strain. The tensile strain of W-BFs/EP composites improved from 9.4% for the neat epoxy to 15.5% for unmodified W-BFs/EP. Compared with the unmodified W-BFs/EP, when the content of KH590, KH570 and KH550 within the epoxy is 6wt%, the maximum enhancements in the tensile strain are 39.3, 76.1 and 67.7%, respectively. Silanized W-BFs/EP significantly increases epoxy's tensile strength in comparison to both pure epoxy and pure BFs/EP.

(Fig. 9c) shows standard cast-in-place stress-strain curves for silanized W-BFs/EP preparation. Adding the silane coupling agent to the epoxy composite makes it more resistant to strain and tensile stress compared to pure epoxy and pure W-BFs/EP composite. Herein, the increased interfacial contact between epoxy matrix and basalt fibers transfers stress from the epoxy to the fibers, improving composite mechanical performance. The basalt fiber is a composite of silicate, which also has same chemical composition with agent of

silane coupling. The agent reacts with the groups of silicon hydroxyl on BF surfaces to generate a new bonding of -Si-O-Si-, which creates a more homogenous silicon-oxide network structure and improves mechanical performance.

The effect of epoxy mixed with various contents and chemical composition of the agent on flexural characteristics of epoxy matrix strengthened by woven BFs are presented in Fig. 10. As shown in Fig. 5a and b, pure W-BFs/epoxy composites had 177.0 and 5.5% higher flexural strength and strain than plain epoxy. Whereas W-BFs/epoxy composites treated with 9wt% KH590, KH570, and KH550 showed a 65.3%, 81.5%, and 74.5% increase in flexural strength, respectively, compared with pure W-BFs/EP composite (271.5 MPa). As the results, it is clear to see that introducing silane molecules could make chemical bonding between the basalt fibers and matrix and improve interfacial interaction than the unmodified composites. Moreover, the interconnected basalt fibers and cross-linked epoxy matrix could assist as a buffer to absorb the failure energy at the interface and hence hinder the cracks propagation and enhance the interfacial bonding resulting in enhancements of flexural strength. Meanwhile, the connection between basalt fibers and epoxy might be compromised due to the lack of silane molecules in the epoxy matrix, perhaps resulting in fissures. In this instance, the basalt fibers were not enough active as a strengthening epoxy, and majority of the external stress was passed through the basalt fibers. Consequently, the risk of delamination between the epoxy matrix and the BFs decreased. Furthermore, the epoxy mixed with silane agents has strong cross-linked among the chains of the resin and supports uniform load transfer capacity of the basalt fibers [19, 39]. As a result, the silanized W-BFs/EP composite possesses better flexural properties than the unmodified W-BFs/EP composites. Interestingly to highlight the bio-improve in the strain of the composites with respect to the brittle epoxy resin. Most up to date strengthen methods, for example, mixes of rubber and polymer can effectively increase durability, but at the expense of mechanical strength [40]. Furthermore, with strong chemical adhesion, the silanized

**Table 2** Mechanical performance of epoxy composites strengthened by woven basalt fibers

Filler type	Method	Increase in flexural strength (%)	Increase in tensile strength (%)	ref
Basalt/GNPs/EP	Hand lay-up and hot press	23.4	10.4	[43]
Basalt-CNTs/EP	Hand lay-up	32.0	-	[44]
Basalt/KH570-CNTs/EP	Hand lay-up	12.5	41.4	[45]
Basalt/KH550-SiO <sub>2</sub> /EP	Hand lay-up	-	30.0	[46]
Basalt/GO/EP	Hand lay-up and hot press	18.0	59.0	[47]
Alkali-basalt/EP	Hot-pressing	5.7	-	[48]
Basalt/KH570-nanoclay/EP	Hand lay-up and vacuum	39.4	9.6	[10]
Basalt/Nano-zirconia/GO/EP	Hand lay-up	50.0	-	[49]
Basalt/KH540-CaCO <sub>3</sub> /EP	Hand lay-up	28.0	20.0	[50]
Basalt/KH570/EP	Cast-in-place	81.2	49.0	This work

W-BFs/EP composite offer extra profits to increase the ductility of composite [41].

Compared with pure epoxy, obviously displayed in Fig. 10b that the flexural strain improved by 93.0, 165.5 and 80.6% with the concentration (6wt%) of KH590, KH570 and KH550, respectively. The addition of silane solution in cascade with S-BFs/epoxy composites significantly enhances crosslinking between the fibers and the matrix. Thus, it is intriguing as the silane coupling agents can make the composites within epoxy more flexibility and increase the flexural strain. The long-chain of silane agents added to pure W-BFs/EP showed the highest flexural strength and strain compared with pure EP/BFs composites. The long-chain silane agents applied to pure W-BFs/EP composites had the maximum flexural strength and strain. The lengthy  $\text{CH}_2$  chains for the coupling agents allowed the silane molecules and BFs to form massive physical bonds that physically interconnected against crack propagation, improving mechanical performance. The  $\text{CH}_2$  flexible and long chains may collaborate closely with the resin, allowing molecules of the silane agent to deeply enter the resin, and activate chemically the silane molecules Si group to connect with the resin and fibers. Bonded agents may change inter-adhesion between BFs and matrix resin. [42]. The typical basalt fibers/epoxy composite flexural stress-strain curves that prepared by impregnating process (shown in Fig. 10c), we can see that adding silane coupling agents to epoxy increases the flexural performance of BFs/epoxy composite compare to pure epoxy and pure of EP/BFs composites.

To further prove our work, the woven basalt fibers/epoxy composites modified by silane coupling agents and prepared via cast-in-placed process were in comparison to those found in other relevant literature, as shown in Table 2. The improvement of tensile and flexural strength for modified woven basalt/epoxy composite by silane coupling agents showed remarkable mechanical performance than the woven basalt fiber/epoxy composites prepared via other techniques and methods as route mentioned in works of literature.

### 3.8 Morphological Analyses of Silanized Epoxy/Woven Basalt Fibers Composites

SEM fracture surface analysis was used to understand how silane coupling agent chemical structure affects W-BFs/EP composite interface behavior. In comparison, the SEM images of fracture surfaces of unmodified W-BFs/EP composite at different magnifications are demonstrated in (Fig. 11a and a1) exhibit the long pull-out basalt fibers indicating a lower bonding of the pure epoxy polymer towards the basalt fiber surface. An adequate silane coupling agents (KH570, KH590 and KH550) into the epoxy structures can give a strong bonding between the epoxy and basalt fibers, as

shown in (Fig. 11b, b1, c and c1). The crack tip was difficult to penetrate through the strong bridge structure formed by silane coupling agents, which could provide effectual stress transfer and damage resistance to external loads. Morphological investigation shows that silane coupling agents with varied chain structures improve basalt fiber-epoxy matrix surface bonding. An enhancement in bonded strength using silane coupling agents is thought to result in physical and chemical bond formation. The epoxy matrix and basalt fibers' strong contact reduces fracture propagation, boosts fracture resistance, and bridges and deflects mechanical stress fractures. Crack-bridging increases energy absorption and minimizes fiber pull-out under mechanical stress, improving composite durability and impact resistance. High-temperature composite structural integrity is maintained by the strong fiber-matrix interface. Basalt fibres' superior adherence to the epoxy matrix and thermal resilience sustain the composite's mechanical qualities at high temperatures.

## 4 Conclusion

The study investigated the influence of silane coupling agents on the toughness of epoxy and woven basalt fiber/epoxy composites. The silane coupling agents (3-mercaptopropyltrimethoxysilane, 3-methacryloxypropyltrimethoxysilane, and 3-aminopropyltriethoxysilane) were mixed with epoxy liquid and studied for their mechanical properties.

Blending 6 wt% of KH560, KH590, KH570, and KH550 into the epoxy enhanced its flexural strength by 69.4, 57.6, 83.4, and 84.7% compared to the pure resin. The modified W-BFs/epoxy composite improved its tensile strength by 77.6, 49.0, and 79.6% compared to the pure W-BFs/EP composite (173.2 MPa). The flexural strength increased by 65.3, 81.5, and 74.5%. The improvement in mechanical performance was due to the creation of physical and chemical interactions with the resin, BFs/EP, and silane coupling agents. The SEM images showed that the cross-linked epoxy chains were stronger and that the surface of the BFs and the resin were better connected. The findings will assist in selecting the best silane coupling agent for basalt-epoxy fiber strength.

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## Declarations

**Ethical Approval** Not applicable.

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