



# Amending the thermo-mechanical response and mechanical properties of epoxy composites with silanized chopped carbon fibers



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## ABSTRACT

This study demonstrates the ability of functionalized chopped carbon fibers (CCFs) chosen from industrial waste to improve the thermo-mechanical properties of CCFs/epoxy composites. The defect sites onto the CCFs were created by their oxidation and the oxidized CCFs were covalently linked with siloxane functional groups to conceal their defects. The surface functionalization of CCFs was characterized by a simple chemical route, FTIR and TGA analysis, respectively. The surface morphology of functionalized CCFs showed the generation of highly dense networked globules. Epoxy composites filled with 0.5 wt% of siloxane attached CCFs (S-CCFs) showed a tremendous enhancement in storage modulus (~376%) without sacrificing their thermal stability. Furthermore, the S-CCFs reinforced epoxy composites demonstrate a significant improvement in the tensile and fracture properties. Such enhancement in the mechanical properties can open up the scope for the utilization of CCFs as a potential cost-effective candidate for high-performance next generation structural composites.

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## 1. Introduction

Chopped carbon fibers (CCFs) have been recently identified as a promising filler material that can be utilized for manufacturing high performance polymer composites [1,2]. The industries prefer epoxy based highly cross-linked polymer networks as a structural composite. This is quite appreciable because they are cost effective and have unattainable advantages like better strength or stiffness to weight ratio, corrosion resistance etc. [3]. Owing to these advantages they are regularly utilized to manufacture carbon fiber reinforced polymer (CFRP) [4] composite materials useful for aerospace, automobile and defense industries. Nevertheless, the restriction still exists for their usage in high-performance engineering applications where durability is the major concern. Durability of such materials is generally governed by the poor capability of the matrix to diminish the unavoidable defects [5]. Attempts have been adopted to omit such drawbacks by introducing nano or micron size fillers in the epoxy network [5–7]. The introduction of nanofillers mainly concerned with the modification of the polymer matrix network by enhancing their volume energy

and to resist new surface generation to suppress the critical stress intensity factor. However, limitations related to cost and complex fabrication procedures suppresses the adaptability of such attempts in industries [8].

The CCFs produced from waste carbon fibers and the products of CFRP industries are highly adopted recently to boost the mechanical and high-temperature properties of thermoplastic polymers [9–11]. Although, CCFs are highly inert and have weak interfacial adhesion [12], which does not hinder the high-temperature properties of the thermoplastics. But, in case of the thermosets like epoxy network, a poor interfacial adhesion of CCFs influentially make them unacceptable to replace the costly nanofillers. To alter the inertness of carbon fibers (CFs), CFRP industries adopt different technique such as oxidation (gaseous, electrochemical, liquid and plasma), surface grafting, and polymer sizing [13,14]. Such attempts are mainly focused on improving the surface interfacial adhesion of CFs with the epoxy network. Owing to the maneuverability and continuity, oxidation of CFs is mostly used to generate active sites by generating oxygen containing functional moieties onto the CFs [15,16]. The surface modification strategy improves the interfacial adhesion through various mechanisms such as chemical bonding, van der Waals bonding, mechanical interlocking and surface wetting between the CFs and the epoxy matrix [13,15,16]. However, many studies illustrate that

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the inherent strength of the oxidized CFs may reduce by 5 to 10% due to the damage in the structural order of outside sheath of the CFs [13,17]. Recently, a non-oxidative chemical functionalization of the CFs studied widely to overcome the drawbacks of the oxidation [18,19]. Free radicals consisting different functional groups from highly reactive intermediates are generally utilized for non-oxidative chemical functionalization [18–20]. Although the non-oxidative treatment of the CFs improves the interfacial properties, but their applicability is restricted to many industries due to a multi-step chemical processing and complex purification process [19,20]. Another alternative is the organophilization of the oxidized CFs with organosilanes that can be an industrial-friendly route to boost the interfacial properties of the oxidized CFs. The carbonaceous surface of CFs oxidized with inorganic acids led to the formation of COOH groups [16,21,22] and shows very good reactivity with organosilanes during the sol-gel chemistry [23]. Moreover, one recent study showed that the inherent strength of CFs can be improved to ~12% by silanization [13]. Thus, the silanization of CCFs might be an effective way to enhance the interfacial adhesion of CCFs with the epoxy network.

In view of the above context, we have used recycled CCFs from the industrial waste CFs and oxidized them to create defects. The oxidized CCFs were silanized and used as fillers to reinforce the epoxy matrix. This study is focused on the investigation of the ability of silanized CCFs to improve the thermo-mechanical properties of the CCFs/epoxy composites.

## 2. Experimental methods

### 2.1. Materials

Industrial waste Polyacrylonitrile (PAN) CFs, class: AS4; Hexcel Composite (France) having high tenacity were recycled to develop CCFs. 3-aminopropyltriethoxysilane 99% (APTES; Alfa Aesar) was used as a silane coupling agent. Dodecylamine 99% (DDA; Sigma-Aldrich) was used to determine oxygenated functional group density. Nitric acid ( $\text{HNO}_3$ ; Merck) was used for oxidation. Toluene was used as reaction medium. Ethanol, acetone, and dichloromethane (DCM; Merck) and double distilled (DD) water were used for cleaning. Diglycidylether of bisphenol-A (Lapox-L12) based epoxy resin and triethylenetetramine (TETA-K6) based hardener (Atul, India) were used to prepare composites.

### 2.2. Synthesis of functionalized CCFs

Recycling of waste CFs to CCFs and their surface functionalization process is schematically shown in Fig. S1 and discussed in Section S1. P-CCFs were oxidized in the presence of  $\text{HNO}_3$ . Initially, 0.3 g of P-CCFs was added into 20 ml of the nitric acid and mechanically stirred (600 rpm) at 80 °C for 1.5 h. After oxidation, the mixture was washed with DD water, ethanol and acetone until the pH value reached to 7, then filtered and dried in an oven at 80 °C for 8 h. The oxidized CCFs (O-CCFs) were subjected to a nucleophilic reaction in the presence of APTES. A blend of 0.3 g of O-CCFs and 25 ml of toluene in a round bottom flask was subjected to an ultrasonic bath sonicator for 1 h. During the sonication, a blend of 5 mL toluene and APTES (1 wt% of O-CCFs) was added drop-wise to the flask. The final mixture was refluxed at 110 °C for 8 h under constant mechanical stirring (600 rpm). The refluxed solution was washed several times with DD water, ethanol and acetone. The final product was dried in an oven at 80 °C for 8 h, which was designated as silanized CCFs (S-CCFs). The sequence of oxidation and silane chemical interactions on the CCF surface is schematically shown in Fig. 1.

### 2.3. Preparation of CCFs/epoxy composites

Generally, the type, quantity, and reactivity of the surface functional elements govern the interfacial interaction between the matrix and fibers. The chemical identity of the interphase elastomers can be realized through the possible mechanism as shown in Fig. 2. P-CCFs/epoxy composites (P-CCFEC) were prepared by blending 0.5 wt% of P-CCFs in an epoxy resin via a high-speed impeller stirring at 1500 rpm followed by vacuum degassing to remove the entrapped air. A curing agent was added in a stoichiometric ratio and mechanically mixed for 5 min at room temperature followed by vacuum degassing. The resulting mixture was poured into a silicon mold and cured in an oven at 80 °C for 2 h. Similar steps were used to prepare neat epoxy (NE), O-CCF epoxy composites (O-CCFEC) and S-CCF epoxy composites (S-CCFEC). The homogeneous and random distribution of CCFs into epoxy matrix was realized from the micrographs of the epoxy composites as shown in Fig. S2 (a–c).

### 2.4. Characterization

The grafting density of acid functional groups onto the CCFs was measured by a simple chemical route as suggested by Marshall et al. [24] and the detail description is given in Section S2. The infrared spectra of CCFs were recorded using a Fourier transform infrared spectrometer (FTIR, Bruker Vertex 80 spectrometer, Germany). The samples in powder form were mixed with a high purity potassium bromide powder and converted to thin films. For the FTIR analysis, the thin films were scanned in the range of 400–4000  $\text{cm}^{-1}$  with a resolution of 0.2  $\text{cm}^{-1}$ . Morphology of the CCFs was characterized by a field emission scanning electron microscope (FESEM, Zeiss, Supra 55VP) at an acceleration voltage of 5 kV. The dynamic mechanical behavior of the samples was studied by a dynamic mechanical analyzer (DMA; 8000 PerkinElmer, USA). The DMA tests of the specimens (dimension:  $25 \times 8 \times 2 \text{ mm}^3$ ) were carried out in a single cantilever mode with an oscillation frequency of 1 Hz. The data was collected from room temperature to 120 °C at a scanning rate of 2 °C  $\text{min}^{-1}$ . Thermal degradation behavior and heat capacity of the samples were determined by a simultaneous thermal analyzer (NETZSCH STA 449 F3 Jupiter, Germany). The degradation studies were carried out at a heating rate of 10 °C  $\text{min}^{-1}$  from room temperature to 800 °C under nitrogen purging gas with a flow rate of 60  $\text{ml min}^{-1}$  and using alumina as the reference material. Heat capacity measurements were carried at a heating rate of 5 °C  $\text{min}^{-1}$  in nitrogen using a sapphire standard for calibration. The tensile and single edge notch bending (SENB) tests were performed according to ASTM D-638 (Type-V) and ASTM D-5045 standards, respectively. The tests were carried out using a universal tensile testing machine (INSTRON, Model 5969, USA) at a crosshead speed of 1  $\text{mm min}^{-1}$ . At the midpoint of the SENB specimen, a sharp notch was machined by a fine band saw. Pre-crack in the notch was initiated by tapping a fresh razor blade. The specific dimensions of SENB specimen are schematically shown in Fig. S3, where crack length ( $a$ ) follow a relation with the width ( $W$ ) of the specimen, such that  $0.45 < a/W < 0.55$ . The critical stress intensity factor ( $K_{IC}$ ) and critical strain energy release rate ( $G_{IC}$ ) were calculated in accordance with the ASTM D-5045 standard. For each composition, at least 5 specimens were tested and the average value is reported. The fracture mechanisms were investigated by an optical microscope (Leica, Model: DM 2500M) and the FESEM.

## 3. Results and discussion

### 3.1. Functional group moieties onto CCFs and their morphology

A simple chemical route as described earlier was used to determine the acidic functional group moieties onto the surface of CCFs.

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