



Silane-modified carbon fibers reinforced cyanate ester/benzoxazine resin composites: Morphological, mechanical and thermal degradation properties



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ABSTRACT

In this paper, various concentrations of silane treated carbon fibers (TCFs) were incorporated within cyanate ester/benzoxazine resin to demonstrate their effects on the morphological, mechanical, thermal decomposition properties of the composites. Mechanical test inferred the distinctive improvements in the values of the flexural strength and modulus. The glass transition temperature (T_g) determined from the DSC measurements were appreciably higher than those of the unfilled blend. TGA results showed an improved in the thermal stability in terms of the $T_{5\%}$, $T_{10\%}$, Y_c , T_{HRI} and LOI for all composites. For instance, the $T_{5\%}$, $T_{10\%}$, Y_c , T_{HRI} and LOI were respectively 345.1 °C, 391.8 °C, 50.1%, 217.1 °C and 37.5% for the composites with 20 wt% of the TCFs. The thermal degradation was assessed by evaluating the variation of the activation energy (E_{ac}) of the composites by using three kinetic models, namely Horowitz and Metzger, Coat and Redfern, and Brodo models. It was found that the E_{ac} greatly increased and further reflected the improvement in the thermal stability of the composites. This could be attributed the unique characteristics of the TCFs that possibly resulted in effective physical barrier effects against thermal degradation, thereby leading to the amelioration of the thermal properties.

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1. Introduction

The unprecedented utility of polymeric blends in recent twenty years has considerably triggered the development of major polymeric technologies into a new and modern era. Polymer blends have chiefly found their uses in myriad applications, making them as one of the most important classes in the field of polymer science and engineering [1]. Blends of cyanate ester (CE) and benzoxazine (BOZ) resins are among those binary systems, which have been attracting during the past few decades a lot of attention from many researchers and investigators [2–9]. Indeed, CE resin has excellent and desirable physical, mechanical and thermal properties when used in room temperature applications. It is relatively stiff and a low melting temperature, low density and relatively resistance to impact [4]. On the other hand, BOZ resin is one of the most

concentrated on polymeric substances and has a wide application in the industry [10]. Because of its suitable mechanical, thermal and dielectric properties, it can be processed easily and thus used in different interesting sectors. Therefore, by combining the aforementioned properties of the homopolymers, several studies reported that the blend of CE and BOZ resins could be considered as super engineering materials due to their attractive properties such as easy processability, good dielectric properties, and high radiation resistance at 500 kGy radiation dose [2–9]. As a result, they could be widely used in electronics, aerospace components and military services [2–9]. However, despite these attractive properties, the CE/BOZ resin blend' potential performance can be limited by their lack of toughness and susceptibility to degradation over the time [11]. Up to this point, there have been relatively few studies on improving the performances of the resin blend. For example, Wang et al. explored the effect of the organic montmorillonite on the properties of the CE/BOZ resin [11]. In the mechanical section, the flexural strength and modulus, which were respectively 118 MPa and 3.86 GPa, were not as much as higher than those of the pure

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resin properties. Another study conducted by Zegaoui et al. when they exposed the blend to various irradiation doses [3]. The obtained results showed that at a 500 kGy irradiation dose, the thermal and mechanical properties were marginally affected by the irradiation dose and a slight enhancement was attained as compared to the un-irradiated resin properties. Consequently, this blend still needs some improvements to meet the industrial requirements and to enlarge their myriad applications, especially where elevated properties are mandatory required.

For further enhanced performances and expanded applications of the resin blend, the reinforcement by using synthetic fibers was doped in this study. Indeed, blending and fiber composite formation techniques are mostly important in the development of new polymeric materials since it is generally accepted that most of raw materials are either of very expensive or poor quality. Carbon fibers (CFs), which are ten times stronger than the steel, are considered as advanced engineering materials [12]. CFs are promising raw materials for fiber reinforced composites [13], especially in high-performance applications due to the fact that they do offer distinguished properties like a low coefficient of thermal expansion, highest specific strength and modulus, of all reinforcing materials along with the other benefits [14]. Consequently, there has been recently an increasing interest in the use of CFs reinforced polymer composites in aerospace, military structural applications [15], renewable energy and other industries [16]. Additionally, CFs are ideal for applications where strength, stiffness, lower weight, and outstanding fatigue characteristics are critical requirements [17]. What is more, they are very applicable for high-temperature resistance, chemical inertness, and high-damping demanding applications [18]. Up to this point, CFs have been used to reinforce several polymer matrices like polyimide [12], polypropylene [14], polytetrafluoroethylene (PTFE) [16], epoxy resin [17], polybenzoxazine [18], cyanate ester [19–21], bismaleimide [22], polyurethane [23] composites and so forth. From these above studies, distinctive enhancements in mechanical properties and thermal stabilities have been effectively attained as compared to the pure polymer matrices. Nevertheless, as per of our knowledge, no attempts till now have been carried out to use CFs as reinforcements for overcoming the brittleness of CE/BOZ resin blends. Therefore, the motivation of this present study was to explore the effects of incorporating various concentrations of CFs on the ultimate performances of CE/BOZ resin matrix.

In this scenario, the CFs were initially treated with the silane coupling agent (SCA) in order to introduce the functional groups that can form a bridge of chemical bonds between the fibers and the resin matrix, and thus enhance to a large extent the dispersion and interfacial adhesion of the fibers since it is widely documented that the fibers function as reinforcing materials only after treatments. Hereafter, a series of polymer composites based on CE/BOZ resin and treated CFs (TCFs) were prepared by a compressing molding technique to create high-performance composite materials with low cost for possible practical applications in various fields. The resultant composites were systematically characterized by means of FTIR, SEM, DSC, flexural and TGA tests. In addition, this current study also focused on understanding the influence of the fibers on the thermal stability, heat resistance index's temperature and flame retardant properties of the composites.

2. Materials and methods

2.1. Materials

A dicyanate ester of bisphenol-A (DCBA) was friendly provided from Institute of Petrochemistry Heilongjiang, Academy of Science (Harbin, China). Bisphenol-A based benzoxazine (BA-a) was

obtained from Jiangxi Huacui Advanced Materials Co. Ltd (China). Shanghai Aladdin Reagents Co. Ltd (Shanghai, China) kindly provided the ethanol, acetone and 3-glycidyloxypropyltrimethoxysilane (GPTMS). Beijing Chuanjing Co., Ltd (Beijing, China) provided the CFs. The CFs have the diameter of 7 μm , length of 6.2 mm, density of 1.76 g/cm^3 , tensile strength of 3500 MPa and Young's modulus of 230 GPa as being reported on the data sheet of the product delivered by the producer. Distilled water was prepared in our laboratory. The chemical structure of the raw materials were depicted in Fig. 1.

2.2. Surface treatments of carbon fibers

In this current study, the authors used the silane technique as it is nowadays the most common methods for fiber surface treatments [12]. Indeed, it has been established that the CFs cannot adhere well when they are used as reinforcement materials in polymer based composites due to the chemically inert surface and low surface energy, which negatively affect the ultimate properties of resulting composites [24]. Thus, tailoring the surface chemistry of CFs consistently allows for the enhancement of interfacial adhesion with a matrix by providing sites for a possible fiber–matrix interaction. The silane treatment could improve to a large extent the computability between the reinforcing phase and the polymer matrix through the establishment of covalent bonds between them [25]. The surface treatment of the as-received CFs was as follow:

- 1) The CFs were washed several times with acetone/ethanol in order to remove the unwanted attached impurities and polarise their external surfaces.
- 2) After washed to neutrality with deionised water, appropriate quantities of CFs were thoroughly blended with 0.9 g of SCA in 60 ml of ethanol and 40 ml of water, and then mechanically stirred using a glass stick for 30 min at a room temperature.
- 3) In order to minimize the adverse effects of the SCA, the resulting CFs were washed with ethanol and distilled water and then dried at temperature for 12 h, and systematically followed by a vacuum drying at 120 $^{\circ}\text{C}$ for 12 h.

Untreated and silane treated CFs were characterised by FTIR spectroscopy to examine the presence of SCA functional groups on the external surfaces of CFs. Indeed, most of the engineers did approve the efficacy of the fiber modifications using SCA from FTIR spectral investigations. Fig. 2 compares the IR spectra of the pure CFs and the modified ones in the range of 4000 to 500 cm^{-1} . As seen from this figure, the IR spectra of the CFs revealed few absorption peaks, which could evidently infer low possibilities of interactions between the CFs and the blend matrix. What is more, these few detected bands might possibly militate against a good fiber/matrix interaction. For example, the broad band located at 3440 cm^{-1} could be attributed to the hydroxyl (O-H) groups that could possibly originate from the water absorbed on the CF surfaces. The frequencies observed at 2940 cm^{-1} and 2840 cm^{-1} could be due to C-H stretching vibration of methyl and methylene groups [26]. The weak band observed at 1715 cm^{-1} could be ascribed to the stretching vibrations of carbonyl groups. The vibrations seen in the region of 1580–1699 cm^{-1} and 1050–1100 cm^{-1} could be assigned respectively to C=C and C-C groups [27]. Note that these detected peaks did ensure the organic character of CFs, which were typical to the most studies related to the CF FTIR studies [28]. However, the FTIR spectra of the TCFs presented some supplementary peaks as clearly observed in Fig. 2. For instance, the frequencies centered at 2940 cm^{-1} and 2840 cm^{-1} could be attributed to the stretching vibrations of (CH_2) groups from the silyl part of the TCFs. The peaks

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