



Functional polycarbonates for improved adhesion to carbon fibre

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ABSTRACT

In order to improve the fibre-matrix interaction in carbon fibre reinforced composites the polycarbonate (PC) matrix polymer was modified by the introduction of ethyl-3,5-dihydroxybenzoate as reactive sequences in the polycarbonate backbone. This promising strategy can be considered as an alternative approach to the modification of the carbon fibre surface to control and tailor the adhesion between carbon fibres and polymer matrix. The modification of the polycarbonate demonstrated improved adhesion to carbon fibre in pressed films, which was observed with microscopy-ATR-FTIR and SEM when compared to unmodified polycarbonate. Single fibre pull-out testing subsequently confirmed the improved adhesion, demonstrating higher interfacial shear strength for the functionalized polycarbonate.

1. Introduction

Polycarbonate resins based on bisphenol acetone (BPA, 4,4'-(*propane-2,2-diyl*)diphenol) have an extraordinary property profile, combining ductility, strength and durability with high transparency and acceptable temperature stability. Materials based on this resin are used in compact discs, appliances, helmets, packaging materials, sunglasses, automotive headlamp lenses, etc. Over the past 50 years, the production and processing of polycarbonate has become a multibillion dollar industry, serving a 3 million tons market in 2009 [1]. Further improvements on mechanical properties like stiffness and strength directed research towards polycarbonate/carbon fibre composites [2,3]; an area of interest shared with many other thermoplastic polymers [4–8].

The role of the interface between the fibre and resin is of great importance from both a processing point of view as well as performance. To exploit the mechanical properties of fibre reinforced thermoplastic composites, the fibre-matrix adhesion must be on an optimized level [9–13]. Different approaches have been followed to increase the adhesion between carbon fibres and polymer matrix that were summarized in detail in Ref. [14]. In general, carbon fibre surface modification is done by wet-chemical (sizing/polymer finish, acidic modification, electrochemical modification), dry-chemical (plasma/high energy irradiation modification, nickel surface coating, thermal modification) and also multiscale methods by applying nano-particles on the surface. For polycarbonate specifically, studies have been done mainly with respect to oxygen plasma-treated carbon fibres [15–17] or electrochemical oxidation [18–21] generally showing significant

increase in adhesion to polycarbonate after treatment.

Modification of polymers for improved adhesion has been studied for numerous thermoset systems, utilizing comonomers as one of the approaches [13,22,23]. For thermoplastic materials, matrix modification is described in literature [15,24–27], as well, but at a much lesser extent. For polycarbonates, the inherent reactivity of the polycarbonate backbone towards reactive sites on the carbon fibre by transesterification was studied [19], as well as the addition of coupling additives which react onto the carbon fibre and scramble with the polycarbonate backbone [27–30], compared to BPA polycarbonate homopolymer [31] (Table 1). Studies on polycarbonate copolymers is focussed on changing material properties like flow, heat and optical performance [32,33], but for improved adhesion to carbon fibres only limited work is presented. Attention to alternative routes was given by studying cyclic oligomers [34].

In this work, we are combining reactive, functional groups, incorporated in the polycarbonate backbone, using a monomer with limited impact on the material properties. The monomer can be reacted by thermal impulse. The selection of functional groups is hindered by the processes of making polycarbonates [32,33], where the melt process requires thermal stability and the interfacial process requires solubility and inertness towards used solvents. Rosenquist [35] has described the use of this reactive group build into the polymer backbone for its use in flame retardant compositions, demonstrating reactivity at elevated temperatures, resulting in network formation. The same principle is studied here for improvement of adhesion to carbon fibre. Multiscale modelling of improvement of mechanical performance of composites is under development [36], currently however, actual

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Table 1
Overview of matrix modifications and micromechanical tests applied on CF/PC composites to increase and characterize the fibre-matrix adhesion by the interfacial shear strength (IFSS); the results represent the lowest and highest achieved value of investigated materials for each reference); *M_w = molecular weight, **SD = standard deviation.

matrix modification				
fibre	matrix	processing conditions	testing method	IFSS
PAN-based CF with unknown sizing (12 K, HTS40, Toho Inc. Corp., Japan) and self-prepared CF with epoxy sizing	PC (Dongguan Plastic Film Corporation China), focusing on polycarbonate backbone transesterification		single fibre fragmentation test	25.04 ± 1.08 MPa (not oxidized); 47.53 ± 1.23 MPa (15 min treatment time) [19]
PAN-based unsized, but commercially surface-treated CF Hercules Magnamite® AS4	Bisphenol-A PC, pure grades: PC16 M _w 16,000 to PC65 M _w 65,000; Commercial grades: Lexan 141 PC	250–300 °C; 8–45 min; 230–275 °C; 15–30 min; 137–1378 kPa	microindentation test single fibre fragmentation test	20.2–48.3 MPa (9–15% SD**) 51.1–56.2 MPa (7–10% SD) [27]
Desized CF supplied by Tae-Kwang Co. (TZ-307)	PC (Sam Yang Co. No. 3022I) M _w 140,000; modified PC as coupling agents: water-dispersible (WDGP) copolymer with long polyacrylamide chains; tetrahydrofuran (THF) -soluble graft copolymers (TSGP) with short polyacrylamide chains	PC processing 240–300 °C; treatment time and temperature with coupling agents: 5–120 min; 40–90 °C	single fibre fragmentation test	Untreated: 25.3 MPa WDGP: 39.8 MPa TSGP: 42.8 MPa [29]
PAN-based Panex 33, Zoltex Rt, electrochemical oxidized	PC (Makrolon 2805, Bayer), MDI, TGI, EPS and SAS coupling agent	PC processing at 280 °C, curing 24 h at 120 °C	fibre fragmentation length	Ranging from 21 to 28 MPa based on coupling agent and amount used [30]
PAN-based CF tape, unidirectional spread (elastic modulus 240 GPa)	Bisphenol A PC, AD5503 (M _w 15,000) and K1300 (M _w 30,000) Teijin Chemicals Ltd., Japan	300 °, 2Mpa, 90sec for consolidation.	single fibre pull out	68.4 MPa (K1300) 53.9 MPa (AD5503) [31]

testing of materials is the preferred method for evaluation of these concepts. In literature, different micromechanical testing methods [36–40] have been established to determine the interfacial shear strength. Table 1 summarizes these techniques. In this work, the single fibre pull-out (SFPO) test is applied to study the fibre-matrix interaction.

2. Materials and experiments

2.1. Materials

PAN (polyacrylonitrile)-based carbon fibre (24 K), unsized, TOHO TENAX® Co. Ltd.

LEXAN™ PC105, a BPA polycarbonate homopolymer, as produced on commercial scale by SABIC, and available as high molecular weight resin for injection moulding. This material was used for lab evaluation, selected for its good melt-strength and ductility.

LEXAN™ HF1110, a BPA polycarbonate homopolymer, as produced on commercial scale by SABIC and available as high flow general purpose grade. This material was used for the SFPO testing, selected for its lower viscosity enabling efficient fibre embedding.

2.2. Synthesis of functional (co)polycarbonate

A 4.9 mol% polycarbonate copolymer of ethyl-3,5-dihydroxybenzoate (EDHB) and BPA (Fig. 1) was prepared according the procedure described by Rosenquist [35]. The resin was isolated as a white powder by steam precipitation followed by drying in a cone shaped vessel using heated nitrogen. From size exclusion chromatography analysis (SEC) (polystyrene standard) of the powder M_w = 48,677 g/mol; P_d = 2.76 was determined. Incorporation of EDHB was confirmed by ¹H NMR (Fig. 3) and verified by ¹³C NMR, the incorporated monomer had a small impact on T_g (measured 141 °C versus 145 °C for the BPA homopolycarbonate).

For the 8.6 mol% polycarbonate copolymer of EDBH and BPA, the same procedure was used, charged amounts were altered as required. From SEC analysis (polystyrene standard) of the powder M_w = 49,585 g/mol; P_d = 2.80 was determined. Incorporation of EDHB was confirmed by ¹H NMR and verified by ¹³C NMR, the incorporated monomer had a small impact on T_g (measured 138 °C versus 145 °C for the BPA homopolycarbonate).

2.3. Characterization

2.3.1. NMR

¹H NMR (proton nuclear magnetic resonance spectroscopy) spectra of both functionalized polycarbonate copolymers were recorded on an Agilent 600 at 600 MHz in CDCl₃ at a concentration of 20 mg/mL with CHCl₃ calibrated at 7.26 ppm: 8 7.90 (d, 2H), 7.50 (t, 1H), 7.25 (d, 4H), 7.17 (d, 4H), 4.40 (q, 2H), 1.68 (s, 6H), 1.39 (t, 3H). The spectrum of the polycarbonate copolymer with 4.9 mol% ester is shown in Fig. 2. The content of EDBH was confirmed for both functionalized polycarbonate copolymers using the integral of the peaks at 7.17 ppm (4H, BPA) and 7.90 ppm (2H, ethyl 3,5-dihydroxybenzoate).

2.3.2. X-ray photoelectron spectroscopy (XPS)

All XPS studies were carried out by means of an Axis Ultra photoelectron spectrometer (Kratos Analytical, Manchester, UK). The

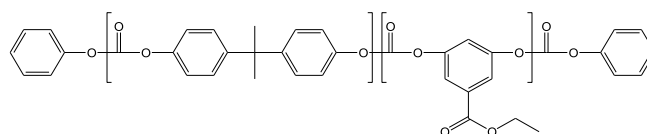


Fig. 1. Idealized structure of EDHB - BPA polycarbonate copolymer.

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