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Original Article

Modification of cyanate resin by conjugated tri-component interpenetrating polymer networks

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ABSTRACT

Cyanate ester (CE) resin was modified in a multi-component way using the technology of conjugated tri-component interpenetrating polymer networks (CTC-IPN). Experimental results showed that the three components in the modified composite existed undependably and they interweaved irregularly in the structure of IPN to form a unique supermolecule structure. The analysis by differential scanning calorimetry (DSC) indicated that the T_g of the modified CE was increased by 22 °C, compared with the T_g prior to modification. When the amount of an initiator was 0.3% or 0.4% and concurrently the ratio of PST/PMMA/CE was 15/15/85, the flexural strength and the impact strength reached the maximum values. Compared with those of pure CE, the flexural strength was increased by 22.65% and the impact strength by 94.99%, respectively.

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1. Introduction

Interpenetrating polymer networks (IPN) is a kind of interweaved net polymer formed by the interpenetration of two or more than two kinds of cross linked polymers [1–5]. This kind of polymer is different from the usual mechanical blending polymer and the graft copolymer. It is the cross linked interaction of different components, interfingering through the polymer network. In general, IPN is not the cross link at the molecular level, but the interpenetration at the supramolecular level. IPN can improve the compatibility of various molecular chains, enhance the network density, and

micronize the phase microstructure to improve the binding force between phase and phase. Thus, the structural changes and phase structure that a single polymer does not have are achieved. The mechanical properties and other properties are eventually improved [5,6].

In 1960, Millar first proposed the concept of IPN [3,7,8]. Thirty years later, Jia Demin, on the basis of this concept, explained the connotation of the conjugated three-component interpenetrating polymer networks (CTC-IPN) [9–11]. He thought that CTC-IPN is a unique ternary interpenetrating polymer network system, of which one polymer is acted as a public network and two other linear polymers, not interpenetrating but contacting closely, penetrate each

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Table 1 – Comparison of mechanical properties between interfacial conjugated inter-penetrating polymers and pure CE under different formulations.

No.	Formulation	CE/MMA/ST (mass ratio)	Impact strength/KJ m ⁻²	Flexural strength/MPa
1	A	75/25/25	10.621	117.611
2	B	80/20/20	12.084	124.152
3	C	85/15/15	15.307	138.626
4	D	90/10/10	11.489	119.064
5	E	95/5/5	10.435	115.096
6	F	100/0/0	7.85	113.023

other and entangle in the public network. This way, three polymers are closely combined to give CTC-IPN composite a special structure, morphology and properties [12,13]. This special interface structure of CTC-IPN is called the interface conjugated interpenetration [7,11,14].

In this work, cyanate ester resin was modified using a CTC-IPN interface conjugated interpenetrating method. Bisphenol A type isocyanate resin (BADCy) was used as a public network to interpenetrate and entangle each other with methyl methacrylate (PMMA) and polystyrene (PST) to finally prepare an interface conjugated three-component interpenetrating polymer composite. The composite prepared was characterized by infrared spectrometer (IRS) and differential scanning calorimetry (DSC). The mechanical properties of the composite were carefully investigated.

2. Experimental

2.1. Experimental raw materials and pretreatment

Bisphenol A cyanate ester resin (industrial product, 98% in purity, white crystal, melting point 79 °C) was made in Ji'nan, China. Prior to experiment, it was dried for subsequent preparation at the temperature between 50 and 60 °C. Methyl methacrylate (MMA), analytical reagent, was made in Tianjin Chemical Reagent Factory No.6, China. It was washed by alkali (0.1 mol/L NaOH) in atmospheric distillation. Styrene (ST), chemical pure, was from Tianjin Dagang Chemical Plant, China. It was washed by alkali (0.1 mol/L NaOH) at atmospheric distillation. Azobisisobutyronitrile (AIBN), chemical pure, was purchased from Shanghai Four Hervey Chemical Co., Ltd. Epoxy resin, bisphenol A type (two glycerine ether DGEBA), epoxy value 0.52, industrial product, was made in Wuxi Di'aisheng Epoxy Co., Ltd.

2.2. Synthesis of interfacial conjugated interpenetrating polymer network

In the presence of trace evocating agent AIBN, MMA and ST were pre-polymerized, respectively, at the temperature between 60 and 70 °C for 2 h. When the reagent turned slightly thickened, a quantitative BADCy melt was added with the temperature around 60 °C. A homogenizer was employed to stir the mixture evenly. The mixture was transferred into a preheated mold and placed in a thermostat of the constant temperature of 70 °C for 1 h. The box was vacuumed to remove air bubbles. According to the curing process of 120 °C/2 h–150 °C/1 h–180 °C/1 h–200 °C/4 h, the mixture was

polymerized and solidified, cooling naturally to room temperature.

2.3. Testing instruments and characterization of performances

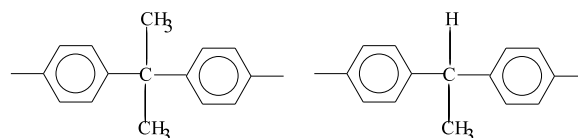
The impact strength and the bending strength were measured, respectively, in German DL-1000B and XCL-40 material testing machines in accordance with the specifications of GB3357-82 and GB3356-82. The deviation was controlled within less than 10%. The sample size was 15 mm × 10 mm × 4 mm. The coating method and the KBr compression method were adopted for infrared spectrum analysis, using a WQF-310 infrared spectrometer made in Beijing Second Optical Instrument Factory. The glass transition temperature was characterized by differential scanning calorimetry in accordance with the national standard GB1634-79. It was measured by a Perkin-Elmer DSC thermal analyzer in nitrogen atmosphere. The starting temperature was 50 °C and the heating rate was increased by 10 °C/min.

3. Results and discussion

3.1. Comparison of mechanical properties of pure CE and interfacial conjugated interpenetrating polymer network

Table 1 indicated the mechanical properties of the modified system under different formulations. As can be seen, except A formula, the impact strength of the PST/PMMA/CE in different mass ratios was all superior to that of pure cyanate ester resin. This is mainly because that cyanate ester resin, methyl methacrylate, and polystyrene interpenetrated each other, entangling to form an interface conjugated interpenetrating network. This network played a role of “forced inclusion” and “synergetic effect” [7,12].

Cyanate ester is a thermosetting resin. Its structural formula is N=C–O–R–C=N, where R stands for



CE was in situ polymerized (curing) into polycyanurate (PCN) at high temperature under the action of catalyst. PCN has many advantages of good toughness, higher glass transition temperature (T_g), lower dielectric constant and loss factor, lower water absorption rate, and inherent flame retardancy. At high temperature (220–270 °C), its molten body is chemically

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