



# Preparation and characterization of UV/thermal dual-curable polyurethane acrylate adhesive for inertial confinement fusion experiment

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## ARTICLE INFO

### Article history:

Accepted 26 November 2015

Available online 5 December 2015

### Keywords:

Adhesive

Polyurethane acrylate

UV/thermal dual-curing

Inertial confinement fusion experiment

## ABSTRACT

A new type of polyurethane acrylate (APUE) oligomer, which contains both acrylic and epoxy group in its molecular structure, was synthesized. UV/thermal dual-curable adhesives were prepared using various contents of APUE oligomers, reactive diluent, photoinitiators and thermal-curing agent. The curing behaviors of the dual-curable adhesives were investigated using photo-DSC, on-line FT-IR spectroscopy, the determination of gel fraction and dynamic mechanical thermal analysis (DMTA). The reaction rate and extent of UV curing were studied and found to be strongly dependent on the concentration of C=C bonds in the APUE oligomers. The gel fraction was also evaluated as a function of C=C bond concentration, demonstrating that gel fraction increased with increasing C=C bond contents in the APUE oligomers. Moreover, when the dual-curable adhesive was thermally-cured, the gel fraction was largely improved, due to the thermal-curing agent initiating the unreacted epoxy groups to allow the formation of further crosslinking. The adhesion strength at  $-196\text{ }^{\circ}\text{C}$  increased with increasing C=C bond content. The latent thermal-curing agent contributed to the enhancement of the adhesion strength.

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## 1. Introduction

Inertial confinement fusion (ICF) is an alternative way to achieving controlled thermonuclear fusion. The basic idea is to ignite and burn a few milligrams of deuterium–tritium fuel by means of high power laser or ion beam pulses. ICF target is one of the key components of ICF facilities; the micro capsule for deuterium–tritium fuel is the core of the ICF target. An adhesive exhibiting a fast curing rate at room temperature, high transparency and good properties at low temperature is required in the ICF target ball sealing [1–2]. As most commercially available low temperature adhesives do not meet the requirements of fast curing at room temperature, the properties of the existing UV-curable adhesives at low temperature are poor [3–4].

The UV-curable adhesive has many advantages, such as fast reaction rates, solvent-free formulations and low energy consumption. Therefore, it has a very broad application prospect in the field of ICF pellet connection, assembly and other special areas.

However, it is difficult to form chemical bonds with glass substrates during its curing process. Also, the UV-curable adhesive has large volume shrinkage when it is cured, which may contribute to large shrinkage stresses within the cured adhesive film [5]. All of the above factors could reduce adhesion strength significantly, to the extent that it may not meet the desired requirements for the ICF target ball sealing process. UV/thermal dual-curable technology has received increasing attention in recent years due to the combination of the functionalities of UV and thermal curing in a curable oligomer-based system [6–8]. So we suggest the UV/thermal dual-curable PUA adhesive could be applied in ICF target ball sealing processes.

Among the prepolymers used for UV-curable adhesives, polyurethane acrylate (PUA) oligomers with acrylic acid functional groups and urethane bonds in the molecular structure have gained increasing attention and rapid development. PUA has a wide range of excellent application properties in combining the properties of polyurethane and acrylate when cured, such as high impact, abrasion resistance, toughness and good cryogenic properties combined with good weather resistance and excellent optical performance [9–12], which have potential applications in ICF target ball sealing applications.

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In this work, we synthesized a new type of polyurethane acrylate (APUE), which simultaneously contains both acrylic and epoxy group in its molecular structure. UV/thermal dual-curable adhesives were prepared using various APUE oligomers, reactive diluent, photoinitiators and thermal-curing agent. The curing process involves UV/thermal dual-cure processes including acrylic photopolymerization and epoxy ring-opening reaction (usually an epoxy ring-opening reaction proceeds much slower than the UV-curing). Their curing behaviors were characterized by photo-DSC, On-line FT-IR spectroscopy, gel fraction determination, together with DMTA and adhesion strength characterization.

## 2. Experimental

### 2.1. Materials

Poly(tetrahydrofuran)(PTMG-1000), Isophorone diisocyanate (IPDI), 2-hydroxyethyl acrylate(HEA), 1,3-Bis(aminomethyl)benzene(m-XDA) and Glycidol(GO) were purchased from Aldrich (USA). Dimethylolpropane triacrylate (TMPTA) was kindly provided by MedicalChem (Yancheng) Manuf. Co., Ltd. Dibutyltindilaurate (DBTDL, purchased from Tianjin Zhongxin Chemtech Co., Ltd., China, without further purification) was used as catalyst. 2-hydroxy-2-methylpropiophenone (Darocur 1173), Kelong Chemical Co, Ltd., (China) was used as a photoinitiator.

### 2.2. Synthesis of acrylate and glycidol-terminated polyurethane (APUE) oligomer

100 g of PTMG-1000, and 44.45 g IPDI were added to a 250 mL three-necked round-bottomed flask equipped with a stirrer, N<sub>2</sub> inlet and a heater. The mixing was carried out at 80 °C. The NCO content was determined by the di-n-butylamine titration method [8]. When the value of NCO reached the theoretical value, the reaction was stopped. When the first step was completed, the temperature was reduced to 40 °C, then the required quantities of GO and HEA were added to the system. The specific proportions for the various constituent reactants are shown in Table 1. When the value of NCO reached 0, full preparation of the APUE was assumed. The various reactions involved in the preparation of APUE are shown in Fig. 1.

### 2.3. Sample preparation

The APUE oligomers were prepared according to the formulation details indicated in Table 1 (samples a–e). With all formulations the relative concentrations of APUE: TMPTA: 1173 photoinitiator were maintained constant at 70:30:3. Mixing the above components was conducted in a paste mixer (provided by Shanghai Sower Mechanical & Electrical Equipment Co. Ltd.) for 10 min at 1500 rpm. The heat generated during mixing was not considered significant. UV curing was performed using a medium

vapor pressure Hg lamp, with radiation intensity on the surface of the sample of 25 mW/cm<sup>2</sup>. The subsequent heat treatment process was performed by putting the UV-cured samples in an oven at 60 °C for 2 h.

### 2.4. Characterization techniques

#### 2.4.1. Fourier transform infrared (FT-IR) spectroscopy

The FT-IR spectra were recorded between 4000 and 400 cm<sup>-1</sup> on a Nicolet 6700 spectrophotometer (Thermo Electron Scientific Instruments Corp., USA) by using potassium bromide (KBr) disks prepared from powdered samples mixed with dry KBr at room temperature.

#### 2.4.2. Photo-differential scanning calorimetry (photo-DSC)

Photo-DSC experiments were conducted using a TA Instruments Q-1000 DSC equipped with a photocalorimetric accessory (Novacure 2100) that use UV/light.

The intensity of the UV light at the sample was 25 mw/cm<sup>2</sup> over the wavelength range of 250–600 nm. The weight of sample was about 7 mg. The sample was loaded into an open aluminum DSC pan with a diameter of 5 mm. Measurements were carried out at 25 °C.

#### 2.4.3. Dynamic mechanical thermal analysis (DMTA)

Dynamic mechanical thermal analysis (DMTA) was carried out on a DMTA 2980 C analyzer (TA Instruments, USA) with double-cantilever mode over a temperature range from 30 °C to 120 °C at a heating rate of 5 °C/min at 1 Hz. All films used for the measurement were cut into strips of approximately 30 mm × 5 mm × 0.15 mm using a razor blade.

#### 2.4.4. Gel fraction

The gel fraction was measured to confirm the crosslinked molecular structure of the dual-curable adhesives after UV and subsequent thermal curing. Films of 5 g were soaked in toluene at 50 °C for 1 day. The insoluble part was removed by filtration and dried at 50 °C until a constant weight was obtained.

#### 2.4.5. Determination of free isocyanate-group content

The determination of the free isocyanate-group content of the systems studied was calculated according to China National Standard HG/T2409-92, using surplus of diisobutylamine reacted with the free isocyanate-groups, and titrated back with HCl. Bromophenol Blue was soluted into the sodium hydroxide solution as a chemical indicator [8].

A medium mercury UV lamp was purchased from Hebei Mechanical and Electrical Equipment Co., Ltd. (Hebei, China) with the intensity of 25 mW/cm<sup>2</sup>.

#### 2.4.6. Adhesion strength

1 g of UV/thermal dual-curable polyurethane acrylate adhesive was placed on the surface of a glass substrate specimen of dimensions of 75 mm × 25 mm × 2 mm which was then placed in contact with another glass substrate to yield an overlap of 25 mm (see Fig. 2). The assembly was cured by using a medium vapor pressure Hg lamp, with radiation intensity on the surface of the sample of 100 mW/cm<sup>2</sup>. The UV-cured assembly was then thermo-cured in an oven at 60 °C for 2 h. Due to the fragile nature of the glass substrates (and hence issues associated with loading and clamping of lap-shear specimens in the testing machine) the substrates were supported by additional aluminium alloy specimens (100 mm × 25 mm × 2 mm) which were bonded onto the glass substrates to produce the specimen arrangement shown in Fig. 2. A room temperature curable commercial epoxy adhesive was selected for this purpose. Following room temperature cure, the supported lap-shear specimens were tested at a machine

**Table 1**  
Compositions of the APUE oligomer.

Sample	GPU (mol)	HEA/GO (mol/mol)	HEA (g)	GO (g)	C=C bonds conc. in the APUE (mol%)
a	0.1	0/2	0	14.82	0
b	0.1	1/3	2.90	5.56	25
c	0.1	1/1	11.61	7.41	50
d	0.1	3/1	8.71	1.85	75
e	0.1	2/0	23.23	0	100

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