



Letter to the Editor

## Viscoelasticity of a vitrified hindered phenol compound during thermal annealing

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**Abstract**

The viscoelasticity of vitrified 3,9-bis{1,1-dimethyl-2[β-(3-tert-butyl-4-hydroxy-5-methylphenyl) propionyloxy] ethyl}-2, 4, 8, 10-tetraoxaspiro [5,5]-undecane (abbreviated as AO-80) during the annealing process was investigated by DMA under the solid shearing model for the first time. The results showed that vitrified AO-80 obtained by quenching from its melting state has super-high damping properties ( $\tan \delta > 26$ ), which are much higher than any polymer materials ( $\tan \delta < 3$ ). Moreover, the vitrified AO-80 annealed at 80 °C under different annealing time has different aggregate structure, resulting in the variation of its viscoelasticity. With the increasing of annealing time the peak value of  $\tan \delta$  decreased, but still higher than any other polymers. These results also provided the evidences of the damping properties improvement of polymer composites added AO-80.

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**1. Introduction**

Hindered phenol compound, 3,9-bis{1,1-dimethyl-2[β-(3-tert-butyl-4-hydroxy-5-methylphenyl) propionyloxy] ethyl}-2, 4, 8, 10-tetraoxaspiro [5,5]-undecane (AO-80, as shown in Fig. 1), was usually applied as a commercial antioxidant of polymers [1]. In our recent studies, it was found that the addition of AO-80 into a wide range of polymers, such as acrylic rubber (ACM), could develop a novel super-damping material [2]. With increasing AO-80 content not only the loss factor increased remarkably ( $\tan \delta > 3.5$ ) but also the peak location was shifted to the room temperature. Our further research indicated that AO-80 was a polymorphous material [3,4]. The commercial AO-80 was highly crystalline, whereas AO-80 obtained by rapid cooling from its melting state was amorphous. This vitrified AO-80 could also be treated into many kinds of aggregate

structure according to different thermal annealing conditions. Applying different aggregate structure of AO-80 into polymer matrix could produce the resulting materials of the different damping properties. For example, the damping property arisen from glass transition of vitrified AO-80 in composite of chlorinated polyethylene (CPE)/AO-80 was much higher than that of CPE [5].

The obtained results manifested that the improvement of polymer damping properties by incorporation of AO-80 may be related to both the original viscoelasticity of AO-80 and the interaction of polymer and AO-80. The latter has been reported in detail in our previous articles [2,5,6]. The present study concentrates on the viscoelasticity of AO-80.

Dynamic mechanical analysis (DMA) was often used to investigate the dynamic viscoelastic property of polymers, while there was no report on applying DMA to characterize viscoelasticity of low-molecular-weight compound like AO-80. The major problem was that AO-80 is very fragile and hard to measure its dynamic viscoelastic property by DMA under the tensile model. To solve this problem, solid

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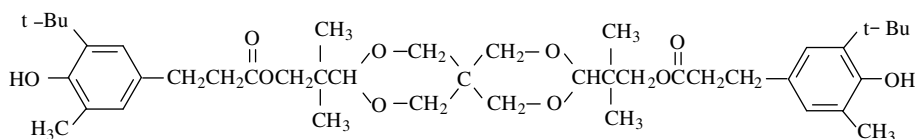


Fig. 1. Chemical structure of AO-80.

shearing model in the characterization of DMA was used to determine the viscoelasticity of different aggregate structure of AO-80. The viscoelastic research of AO-80 would be developed into a new method to design not only the high-damping property material but also the other functional materials. It was also significant to fill the blank of the viscoelastic research of other organic low-molecular-weight compounds.

## 2. Experimental

### 2.1. Materials

The initial AO-80 (ADK STAB, Asahi Denka Industries Co., Japan) is highly crystalline and the chemical structure is shown in Fig. 1. Vitrified AO-80 was obtained by means of melting initial AO-80 at 140 °C for 30 min and then quenching in an ice-water bath.

### 2.2. Thermal annealing of AO-80

The annealing processes were carried out in the oven at 80 °C. Vitrified AO-80 laid onto the metal film was put into the oven for various time intervals, and then promptly quenched into an ice-water bath. After cooling for around 5 min, the samples were taken out and dried in the air.

### 2.3. Characterizations

Differential scanning calorimetry (DSC) (Shimadzu TA-50WS) was used to detect the effects of annealing time on crystallization behavior of amorphous AO-80. About 7 mg weight samples sealed in an aluminum crucible were heated from –20 to 150 °C at a scanning rate of 10 °C/min under nitrogen atmosphere.

The viscoelasticity of AO-80 was analyzed by dynamic mechanical analysis (DMA) (U.B.M. RheologyE4000, Japan) under solid shearing model. The sample specimens whose dimension was 6 × 6 × 2 mm were heated at a constant frequency of 110 Hz and a heating rate of 1 °C/min in a range of 40–120 °C.

## 3. Results

### 3.1. Thermal behavior of AO-80 during annealing

The thermal properties of AO-80 annealed at 80 °C are shown in Fig. 2. The initial AO-80 exhibited a large endo-

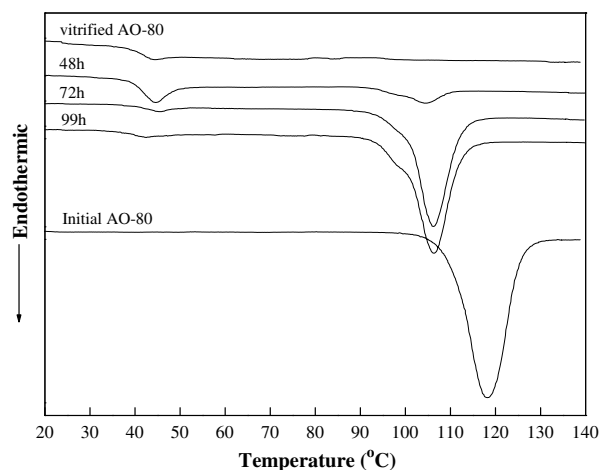


Fig. 2. The DSC curve of vitrified AO-80 at different annealing time.

thermic peak at 119 °C because of the melting of crystalline and  $\Delta H = 97$  J/g. While the melting peak of vitrified AO-80 disappears, a second-order transition appears at about 42 °C deemed to be the glass transition of AO-80. The vitrified AO-80 annealed at 80 °C for 48 h shows a small endothermic peak at 109 °C above the glass transition temperature and below the melting point of initial AO-80. With further increasing of the annealing time, the glass transition trends to be smaller, while the melting peak becomes larger and larger. However, the location of the melting peak is unchanged. Different aggregate structures may lead to different viscoelasticity, so the viscoelasticity of AO-80 annealed at 80 °C was monitored by DMA.

### 3.2. Viscoelasticity of vitrified AO-80 during annealing

The viscoelasticity of vitrified AO-80 determined by DMA are shown in Figs. 3 and 4. Fig. 3 shows the temperature dependence of the storage modulus  $G'$  for vitrified AO-80 during annealing processes. Clearly, the modulus of vitrified AO-80 decreases promptly after glass transition, indicating AO-80 has been in flowing state. The cases for the vitrified AO-80 annealed for more than 48 h are interesting. It is found that a new and relatively wide modulus plateau like polymers after glass transition appears. Meanwhile, the plateau has become higher and covered much wider temperature range with the annealing time increased. The increasing of  $G'$  may be arisen from the increasing of crystallinity as shown in the DSC results.

As can be seen from Fig. 4, the loss tangent ( $\tan \delta$ ) of vitrified AO-80 without annealed sharply increases to 26

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