

**CHAPTER IV**  
**Studies on Miscible Blend of ESCOR™ 325 Terpolymers and EAA**  
**Copolymers: Rheological, Thermal and Mechanical Properties.**

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**ABSTRACT**

Polymer blending is a very useful way to develop materials with certain desirable properties. Synergistic properties often result when components are truly miscible. Blends of Escor™ 325 terpolymers and four different grades of EAA copolymers containing different amount of acrylic acid at various blend compositions were prepared by melt mixing in a twin-screw extruder. The miscibility of blends was investigated using dynamic mechanical measurements. Rheological, thermal, mechanical, and dynamic mechanical properties of the Escor™/EAAs blends were also studied. The results showed that rheological properties, such as storage and loss modulus, of the blends slightly increased with increasing EAA content, but the complex viscosity of the blends decreased with increasing EAA contents. In addition, most blends exhibited improvements in tensile strength at break, Young's modulus, hardness (Shore-D), and a reduction in elongation at break with increasing EAA content. Escor™/EAA5 blends containing between 80 and 95%wt of EAA1 and EAA5 showed synergistic behavior (tensile strength, Young's modulus and hardness) due to higher percent crystallinity, whereas most blends with low EAA contents showed property values below a linear relationship because of phase separation.

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## 1. INTRODUCTION

During the last 10 years remarkable advances have been made in the area of polymer blends both scientifically and technologically. This is manifested by growing number of monographs, publications and from the introduction of new commercial polymeric materials [1]. A polymer blend is basically a mixture of two or more different polymers. The preparation of polymer blends is often a very useful way to develop materials with certain desired properties such as improved toughness, better processability, or better temperature and chemical resistance. In addition blending can usually be implemented more rapidly and economically than development of new chemistry. Therefore, the use of polymer blends is becoming an important factor in the needs of specific sectors of the polymer industry owing to economic incentives [2].

When two polymers are blended, it can result in either a miscible blend or an immiscible blend. Blending depends on thermodynamics of mixing [3]. If the free energy of mixing is positive ( $\Delta G_{\text{mix}} > 0$ ), the blending will result in immiscible blend with the polymer components as separate phases and poor adhesion between the phases. If the free energy of mixing ( $\Delta G_{\text{mix}} < 0$ ) is negative then the polymer blend is miscible and consists of a single homogenous and thermodynamically stable phase. Moreover the formation of miscible blend requires the presence of favorable interaction between the component polymers. The miscibility is measured by numerous techniques such as transparency, microscopic and glass-transition temperature, etc.

Ethylene-acrylic-acid copolymer (EAA) is an ethylene-acrylic acid random copolymer. EAA copolymer is a soft, tough, transparent and similar to low density polyethylene (LDPE). Its attached functional groups provide specific interaction with other polymers such interaction between the neutralizing hydrogen of the acid group with a carbonyl oxygen of another acid group. Thus, EAA copolymer are popular as one part of blend [4]. For example with polyamides 6 [5], polyacetals[6] and epoxidized natural rubber [7].

This research studied the miscibility of blend of ESCOR<sup>TM</sup> acid terpolymer with different grades of ethylene-acrylic acid (EAA1 – EAA5) copolymers. Both

polymers (ESCOR<sup>TM</sup> and EAAs) contain acrylic acid groups, and it is thought that hydrogen-bonding interactions between these groups would likely lead to substantial miscibility. Rheology, morphology and dynamic mechanical properties of the blends were also studied. One possible application of the ESCOR<sup>TM</sup>/EAAs blends is in vibration damping system.

## **2. EXPERIMENTAL DETAILS**

### **2.1 Materials**

Four different grades of Ethylene Acrylic Acid copolymers (EAA1, EAA2, EAA4 and EAA5), containing different amount of acrylic acid, were employed in this study. Ethylene-Methyl Acrylate-Acrylic (E-MA-AA) was similar commercial materials under trademark ESCOR<sup>TM</sup> in areas outside of North America. The grade of ESCOR<sup>TM</sup> employed in this study was ESCOR<sup>TM</sup> 325 that obtained from Exxon Mobil Chemical. The composition of four grades of EAA copolymers and ESCOR<sup>TM</sup> 325 terpolymers are summarized in Table 1.

### **2.2 Polymer Blend Preparation**

ESCOR<sup>TM</sup> 325 terpolymers and EAA copolymers (EAA1, EAA2, EAA4 and EAA5) were dried in a hot air oven at 50°C for 1 day prior to blending. A Collin co-rotating twin screw kneader ZK-25 at 50 rpm and zone temperature either 125°C or 130°C was used to prepare blend of ESCOR<sup>TM</sup> 325 and EAAs. The polymer melt was solidified in chilled water (temperature ~ 25°C) and palletized. The pellets were dried in as a hot air oven at 60°C for 1 days and kept in sealed plastic bags. The samples were later compression molded using a Wabash V 50H compression press machine at 160°C for 5 min and under a force of 10 tons at 160°C for 3min. The samples were denoted according to their weight/weight composition: 0/100, 5/95, 10/90, 20/80, 30/70, 40/60, 50/50, 60/40, 70/30, 80/20, 90/10, 95/5, and 100/0 ESCOR<sup>TM</sup> 325/EAA blends.

### 3.3 Rheological study

In order to compare viscosity and elasticity ratios of ESCOR™ 325 terpolymers to EAA copolymers, the complex viscosity ( $\eta^*$ ), storage modulus ( $G'$ ) and loss modulus ( $G''$ ) of ESCOR™ 325 terpolymers and four different grades EAA copolymers as a function of angular frequency ( $\omega$ ) were measured by an ARES Rheometric Dynamic Analyzer RDA-II with cone-and-plate geometry ( $R=12.5$  mm), in the dynamic mode. The frequency sweep test were carried out at  $130^\circ\text{C}$  and to make sure that the behavior of the test specimens were in linear viscoelastic range, frequency and strain amplitude were varied in the range between 0.1 to 100 rad/s to obtain a suitable range of the torque.

### 2.4 Differential Scanning Calorimetric (DSC) study

DSC studies of pure components as well as of the blends were performed using a Perkin-Elmer DSC7 using nitrogen gas as a purge gas. The sample of  $9 \pm 0.1$  mg was heated from  $30^\circ\text{C}$  to  $140^\circ\text{C}$  at heating rate of  $5^\circ\text{C}/\text{min}$  and cooled down to  $30^\circ\text{C}$  at cooling rate of  $5^\circ\text{C}/\text{min}$  and reheated it again from  $30^\circ\text{C}$  to  $140^\circ\text{C}$  at heating rate  $5^\circ\text{C}/\text{min}$ . The first heating scans were carried out to erase any thermal history of the sample.

### 2.5 X-ray diffraction study

X-ray diffraction patterns of samples were obtained using a Rigaku Rint2000 diffractometer equipped with a graphite monochromator and a Cu tube for generating a  $\text{CuK}\alpha$  radiation ( $1.5046 \text{ \AA}$ ). First, sheet samples were erased their history of polymers using DSC at heating rate  $5^\circ\text{C}/\text{min}$ . Then, the sheet sample was put on a glass slide specimen holder. The sample was examined between  $5^\circ$ - $35^\circ$   $2\theta$  at a scanning rate of  $2^\circ$   $2\theta$  /min in  $0.02^\circ$   $2\theta$  increments.  $\text{CuK}\alpha$  radiation with  $\lambda = 0.154$  nm was used as the X-ray source and operated at 40 kV and 30 mA.

### 3.7 Mechanical study

The tensile properties of the blend were determined at room temperature according to ASTM D1708 method using a Universal test machine (Instron Co.: Model 4206). At least five specimens per blend sample were tested using a crosshead speed of  $130\text{mm min}^{-1}$ . A shore D durometer was used to measure hardness of the blends according to the ASTM D2240 method.

## 4. Results and discussion

### *Rheological property*

As the processing of polymer in most cases involve flow of the material. There are two fundamental rheological properties that are of primary concern in polymer processing. These are viscous and elastic properties [8]. The viscosity is a very useful and revealing rheological function that describes a material in the molten state [9]. Fig 1(a-d) show the complex viscosity ( $\eta^*$ ) as a function of frequency. The complex viscosity value ( $\eta^*$ ) of the ESCOR<sup>TM</sup> 325/EAA blends are in between pure ESCOR<sup>TM</sup> 325 and EAAs. The complex viscosity ( $\eta^*$ ) of the blends decreases with increasing frequency, indicating pseudoplastic nature of the blends. This occurs because the applied force disturbs the long chains of the polymer from their equilibrium position and the molecules become disentangled in the direction of the force causing a reduction in viscosity [8]. The  $G'$  and  $G''$  of ESCOR<sup>TM</sup> 325/EAA blends lie between values of  $G'$  and  $G''$  for pure ESCOR<sup>TM</sup> 325 and pure EAAs (see in Fig 2(a-d) and Fig 3(a-d)). The  $G'$  and  $G''$  of the pure EAA copolymers is higher value than that pure of ESCOR<sup>TM</sup> 325 terpolymer. Therefore, when EAA is added to Escor<sup>TM</sup>325, the  $G'$  and  $G''$  of the blend slightly increases with increasing amount of EAA content. However, the  $G'$  and  $G''$  of the blends at high frequencies does not change much with addition amount of EAA content, indicating that for the storage modulus and loss modulus during mixing of pure materials and polymer blends are almost the same at high frequencies. Moreover, It is also apparent that  $G'$  increases with increasing frequency; indicating that there is an increase in the stiffness of polymer blend

### *Thermal property and % crystallinity*

To understand the thermal property of ESCOR™ 325 terpolymer and EAA copolymer blends, differential scanning calorimetric studies of the pure components of ESCOR™ 325, EAA1, EAA2, EAA4, EAA5 and their blends were carried out in the temperature range 30 to 140°C. The peak temperatures for these samples are listed in Table 2. These values are relatively close to the published works [4,10]. The comparison the effect of % acrylic acid to the crystallization temperature of the pure components. EAA1 copolymer has the lowest % acrylic acid that crystallizes at the highest temperature than those pure components. It might be explained that the minor monomer of a random copolymer or terpolymer acts as an impurity, which in turn acts as a crystal defect. If the minor monomer content in the polymer chain is large, thinner thickness of crystals will result having low crystallization temperature [12]. In terms of the melting temperature, the melting temperature depends on thickness of crystal form during the cooling condition and total defect content of forming crystal are also affects the crystallization temperature [12] or alternatively the melting temperature is an inversely function of acrylic acid content. [13]. ESCOR™ 325 and EAA1 show the lowest and the highest melting temperature at 69.9°C and 100.5°C, respectively. EAA1 probably has the thickest of crystal thickness corresponding to the result of lowest amount of defect content of forming crystal whereas ESCOR™ 325 probably has the thinnest of crystal thickness. EAA2, EAA4, and EAA5 showed intermediate values with EAA1 and ESCOR™ 325. This appearance is also relates to their crystallization results.

For the crystallization thermogram of ESCOR™ 325/EAA1 blends is shown in figure 4a. The upper and lower curves represent the crystallization peak of EAA1 and Escor™ 325, and intermediate curves are blends containing 20, 40, 50, 60, and 80%wt EAA1 content. The crystallization temperatures of the blends exhibit double crystallization temperatures at 20, 40, 50 and 60%wt EAA1 content. This observation might be due to phase separation or possibly artifacts from blending efficiency. A single crystallization peak could be observed at 80% EAA1 blend, and observed peak appeared at higher crystallization temperature than those of pure components. These observations suggest that there is a possible co-crystallization of the blends, caused by increase in interaction between blend components of these

blends or crystallization of two crystalline pure components in the same lamellae crystals [14]. Figure 4(b-d) illustrates typical crystallization peaks of ESCOR™ 325/EAA2, EAA4 and EAA5 blends. The results obtained showed similarity to that of ESCOR™ 325/EAA1 blends. The blends presented the double crystallization peaks at 20, 40, 50 and 60% wt of EAA2, EAA4 and EAA5, but showed a single crystallization peak at high %EAA2, EAA4 and EAA5 (over 80%wt) content.

In case of the melting temperature of ESCOR™ 325 blends with EAA1, EAA2, EAA4 and EAA5 blends. The melting temperature of the blends is plotted as a function of the %EAA content in the blends as illustrated in Fig 5(a-d). The melting temperature of ESCOR™ 325/EAA1, EAA2, EAA4 and EAA5 blends at 20, 40, 50, and 60%wt EAA content exhibits two melting peaks corresponding to the melting temperature of their constituents. These observations illustrated the bimodal distribution of crystal thickness, i.e. the polyethylene from ESCOR™ 325 terpolymers form a separate crystalline phase to the polyethylene in EAA copolymers. However, The melting temperature of Escor™ 325/EAA1, EAA2, EAA4 and EAA5 blends at 80%wt EAA content presented a single melting peak at a slightly higher melting temperature than those of pure components. This could be explained by the polyethylene segments from the terpolymer and copolymer are mixed together and/or some interaction between the components is presented as reflected by the shift to higher temperature than that of the pure components [15]. The results of melting temperature measurements are shown in Table 3.

It was difficult to accurately determine the crystallinity measurements from baseline determination of DSC curves. Thus, the fractional crystallinity of the blends were determined via x-ray crystallography experiments, where thermal history of the samples were erased in the DSC experiment. It is also important to point out that the crystallinity results from DSC and XRD measurements were not different for all measurements [4]. From x-ray crystallography of ESCOR™ 325, EAA5 and ESCOR™ 325/EAA5 blends (Fig 6), there are two sharp reflections at  $2\theta$  values of  $21.4^\circ$  and  $23.7^\circ$ , which can be assigned to the 110 and 200 reflections of orthorhombic sub cell. The amorphous halo is centered on  $2\theta = 20.63$ . D-spacing of the unit cell and  $2\theta$  value of ESCOR™ 325, EAA5 and ESCOR™ 325/EAA5 blends

are almost the same. The X-ray diffraction data indicate that there is no detectable modification of the crystal structure of the components in the blends, as compared to those observed separately [16]. This observation illustrates that the two components of the mixtures crystallize at the same position. This behavior is similar to blends of ESCOR<sup>TM</sup> 325 with other EAAs. The percent crystallinity of the pure components and their blends from x-ray crystallography experiments are shown in Table 3. The percent crystallinity of ESCOR<sup>TM</sup> 325/EAA blends gradually increased with increasing EAA content. These results could be used to explain the effects of blending on the mechanical properties of the blends.

#### *Mechanical properties*

Tensile test is the most widely employed test to characterize the mechanical properties of material. Fig 7(a-c) shows the tensile properties of ESCOR<sup>TM</sup>325 and EAA1-EAA5 blends of different compositions. All blends showed increasing tensile strength at break and Young's modulus (Fig. 7(a-b)) with increasing amount of EAA copolymers. In addition to the tensile strength and Young's modulus of the EAA1 and EAA5 blends are found that there is a synergism at high (over 80% EAA content) EAA1 and EAA5 content. These results supported observations from DSC and XRD i.e., co-crystallization were observed at high EAA content blends. This could be explained by the greater interaction between the blend constituents as suggested earlier [7]. However, results from the elongation at break were relatively different. The elongation at break of the blends was slightly decreased with the addition of EAA content (Fig 7c). For the hardness test results, presented in Fig 7, indicates that hardness monotonically increases as EAA content increases. It means that the blends have high resistance to deformation or to abrasion with increasing EAA content. The synergistic behaviors are shown in the blends made with EAA4 and EAA5 at high EAA content. This behavior could probably be due to the high fractional crystallinity of these blends as suggested in the earlier discussion on the x-ray diffraction experiment.



### *Miscibility study*

The miscibility in the molten state of the ESCOR™ 325/EAA5 blends was determined using the log-additivity rule [9]. Because the composition dependence of viscoelastic functions gives much information about the degree of miscibility of the blend material.

$$\text{Log } F_b = \phi_m \log F_m + \phi_d \log F_d$$

Where  $F$  is a viscoelastic function,  $\phi$  is a volume fraction and subscripts “b”, “m” and “d” indicates the value of the blend, the matrix and the disperse phase, respectively. On the other hand, the viscoelastic functions for immiscible blends deviated from the log-additivity rule. Immiscible polymer blends can be classified into three categories, depending on the blend composition dependence of the viscoelastic function. These categories as (i) positive deviation, (ii) negative deviation, and (iii) positive-negative deviation depending on whether the deviation from the log-additivity rule is positive, negative or both in different composition regions.

Figure 8 shows the complex viscosity,  $\eta^*(\omega)$ , of the ESCOR™ /EAA5 as a function of the EAA5 content at various frequencies. The relation between  $\eta^*(\omega)$  and EAA fraction of ESCOR™ 325/EAA5 blends showed slightly positive deviation from the log-additivity rule at EAA content between 0 and 40% EAA5, while the experimental data lied on the data of log-additivity rule occurs for EAA 5 content greater than 40% EAA5 content. It indicates that this type of behavior the typical for miscible blends. It also clearly took place in these ESCOR™ 325/EAA5 blends, where the chemical nature of the compared blends are very similar, and most of processing parameters are the same.

## 5. Conclusions

This work focuses on the study of rheological, mechanical and thermal properties and miscibility study of ESCOR™ 325 terpolymers and four different grades of EAA copolymers. The results from the rheological measurement indicate that the addition of EAA copolymers in the blends led to the increased of the rheological properties. The complex viscosity ( $\eta^*$ ) of the blends was decreased with increasing frequency, and the storage modulus ( $G'$ ) and loss modulus ( $G''$ ) were slightly increased with increasing frequency.

From thermal analysis, the crystallization curves of blends of ESCOR™ 325 and four different grades of EAA at 60, 50, 40, and 20 %wt of EAA content, They showed two crystallization peaks, which is probably due to the different crystallization rate between ESCOR™ 325 and EAA copolymers. However, the crystallization peaks of these blends were higher than those of the pure components, this may be due to some interaction between two components. These results corresponded to results obtained from melting temperature results. Crystallization curves of ESCOR™ 325/EAA1, 2, 4 and 5 blends at over 80%wt EAA content showed only a single crystallization temperature. This was because of co-crystallization and was corresponded to their melting temperatures.

Mechanical properties of blends of ESCOR™ 325/EAA5 such as Young's modulus and tensile strength at break and hardness were increased with increasing EAA content. However, the elongation at break results were decreased with increasing EAA content. The ESCOR™ 325/EAA5 blends at over 80% EAA content showed synergistic behavior, due to higher percent crystallinity (probably caused by co-crystallization). This is because both materials can be crystallized and form stronger interaction between themselves.

The miscibility in molten state of ESCOR™ 325/EAA5 was observed at EAA5 content greater than 40%. It might suggest that the chemical nature of the components was very similar, and most of processing parameters was the same.

## 6. ACKNOWLEDGMENTS

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**CAPTION OF FIGURES**

- Figure 1a** Complex viscosity,  $\eta^*$ , as a function of frequency for pure EAA1, pure ESCOR™ 325 and ESCOR™ 325/EAA1 blends.
- Figure 1b** Complex viscosity,  $\eta^*$ , as a function of frequency for pure EAA2, pure ESCOR™ 325 and ESCOR™ 325/EAA2 blends.
- Figure 1c** Complex viscosity,  $\eta^*$ , as a function of frequency for pure EAA4, pure ESCOR™ 325 and ESCOR™ 325/EAA4 blends.
- Figure 1d** Complex viscosity,  $\eta^*$ , as a function of frequency for pure EAA5, pure ESCOR™ 325 and ESCOR™ 325/EAA5 blends.
- Figure 2a** Storage moduli,  $G'$ , as a function of frequency for pure EAA1, pure ESCOR™ 325 and ESCOR™ 325/EAA1 blends.
- Figure 2b** Storage moduli,  $G'$ , as a function of frequency for pure EAA2, pure Escor™325 and Escor™325/EAA2 blends.
- Figure 2c** Storage moduli,  $G'$ , as a function of frequency for pure EAA4, pure ESCOR™ 325 and ESCOR™ 325/EAA4 blends.
- Figure 2d** Storage moduli,  $G'$ , as a function of frequency for pure EAA5, pure Escor™325 and Escor™325/EAA5 blends.
- Figure 3a** Storage moduli,  $G''$  as a function of frequency for pure EAA1, pure ESCOR™ 325 and ESCOR™ 325/EAA1 blends.
- Figure 3b** Storage moduli,  $G''$ , as a function of frequency for pure EAA2, pure ESCOR™ 325 and ESCOR™ 325/EAA2 blends.
- Figure 3c** Storage moduli,  $G''$ , as a function of frequency for pure EAA4, pure ESCOR™ 325 and ESCOR™ 325/EAA4 blends.
- Figure 2d** Storage moduli,  $G''$ , as a function of frequency for pure EAA5, pure ESCOR™ 325 and ESCOR™ 325/EAA5 blends.
- Figure 4a** DSC crystallization peaks of pure EAA1, ESCOR™ 325 and ESCOR™ 325/EAA1 blends containing 20, 40, 50, 60 and 80%wt EAA1.
- Figure 4b** DSC crystallization peaks of pure EAA1, ESCOR™ 325 and ESCOR™ 325/EAA2 blends containing 20, 40, 50, 60 and 80%wt EAA2.

- Figure 4c** DSC crystallization peaks of pure EAA1, ESCOR™ 325 and ESCOR™ 325/EAA4 blends containing 20, 40, 50, 60 and 80%wt EAA4.
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- Figure 5a** DSC melting peaks of pure EAA2, ESCOR™ 325 and ESCOR™ 325/EAA1 blends containing 20, 40, 50, 60 and 80%wt EAA1.
- Figure 5b** DSC melting peaks of pure EAA2, Escor™325 and ESCOR™ 325/EAA2 blends containing 20, 40, 50, 60 and 80%wt EAA2.
- Figure 5c** DSC melting peaks of pure EAA2, Escor™325 and ESCOR™ 325/EAA4 blends containing 20, 40, 50, 60 and 80%wt EAA4.
- Figure 5d** DSC melting peaks of pure EAA2, Escor™325 and ESCOR™ 325/EAA1 blends containing 20, 40, 50, 60 and 80%wt EAA4
- Figure 6** X-ray diffraction patterns of ESCOR™ 325/EAA1 blends : (a) pure ESCOR™ 325, (b) 20% EAA1, (c) 40% EAA1, (d) 50% EAA1, (e) 60% EAA1, (f) 80%EAA1, and (g) pure EAA1.
- Figure 7a** Young modulus of ESCOR™ 325/EAAs blends.
- Figure 7b** Tensile strength at break of ESCOR™ 325/EAAs blends.
- Figure 7c** Elongation at break of ESCOR™ 325/EAAs blends.
- Figure 8** Hardness of ESCOR™ 325/EAAs blends
- Figure 9** Complex viscosity,  $\eta^*(\omega)$  as a function of blend composition for ESCOR™ 325/EAA5 blends at various frequencies

**Table 1** Copolymer and terpolymer compositions (all values are in mole fractions)

Sample	Ethylene	Acrylic Acid	Methyl Acrylate
Escor <sup>TM</sup> 325	0.893	0.028	0.079
EAA1	0.988	0.012	-
EAA2	0.974	0.026	-
EAA4*	0.974	0.026	-
EAA5	0.961	0.039	-

\*EAA4 has a lower average molecular weight than EAA2

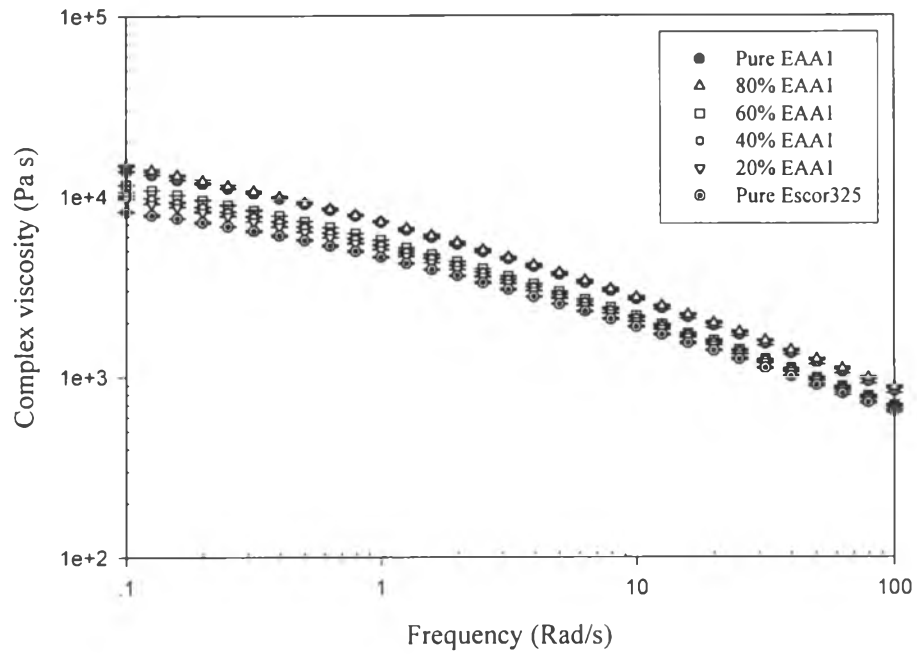
**Table2** The melting and crystallization temperature of pure components

Sample	T <sub>m</sub> (°C)	T <sub>c</sub> (°C)
Escor <sup>TM</sup> 325	69.93	50.73
EAA1	100.52	87.23
EAA2	97.18	83.48
EAA4	96.52	82.15
EAA5	93.43	76.07

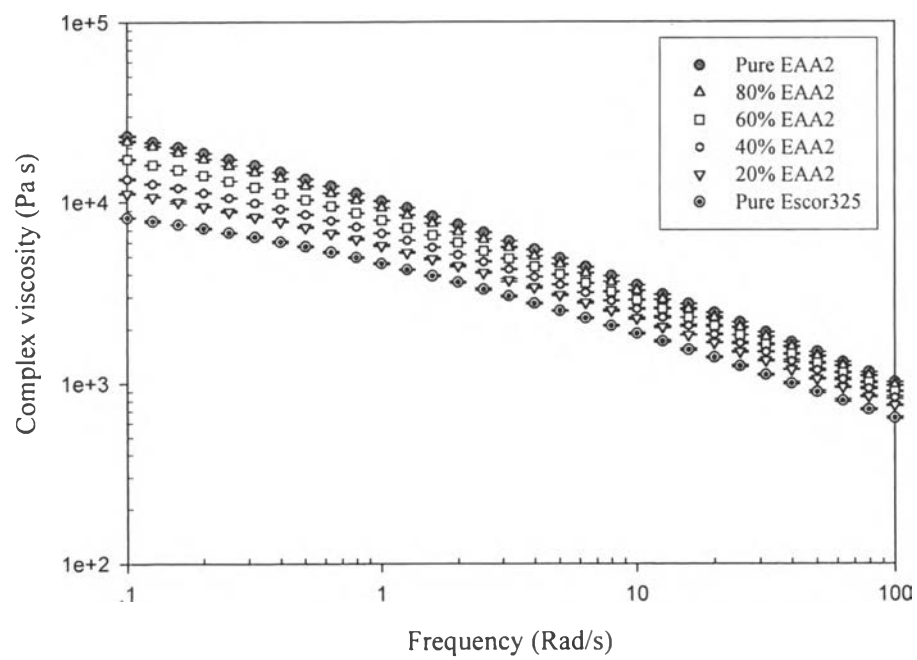
**Table 3** The melting temperature and % crystallinity of ESCOR™ 325 with EAA1, EAA2, EAA4 and EAA5 blends

EAA (%wt)	EAA1		EAA2		EAA4		EAA5	
	Tm (°C)	% crystallinity	Tm (°C)	% crystallinity	Tm (°C)	% crystallinity	Tm (°C)	% crystallinity
0	69.93	5.71	69.93	5.71	69.93	5.71	69.93	5.71
20	70.10, 100.35	10.98	63.02, 98.10	10.53	71.68, 96.83	10.94	62.93, 93.18	9.05
40	68.60, 100.52	12.84	61.35, 97.43	12.60	75.35, 96.77	11.87	62.93, 93.18	9.45
50	69.85, 100.60	13.84	65.35, 97.60	12.78	96.85, 96.85	12.60	62.93, 93.10	9.81
60	68.10, 100.52	13.94	66.60, 97.68	13.36	96.93, 96.93	12.75	58.93, 93.10	10.44
80	101.02	21.89	72.60, 98.27	15.41	97.27, 96.27	16.45	93.52, 93.35	11.24
100	100.52	21.77	97.18	17.96	96.52	16.40	93.43	11.94



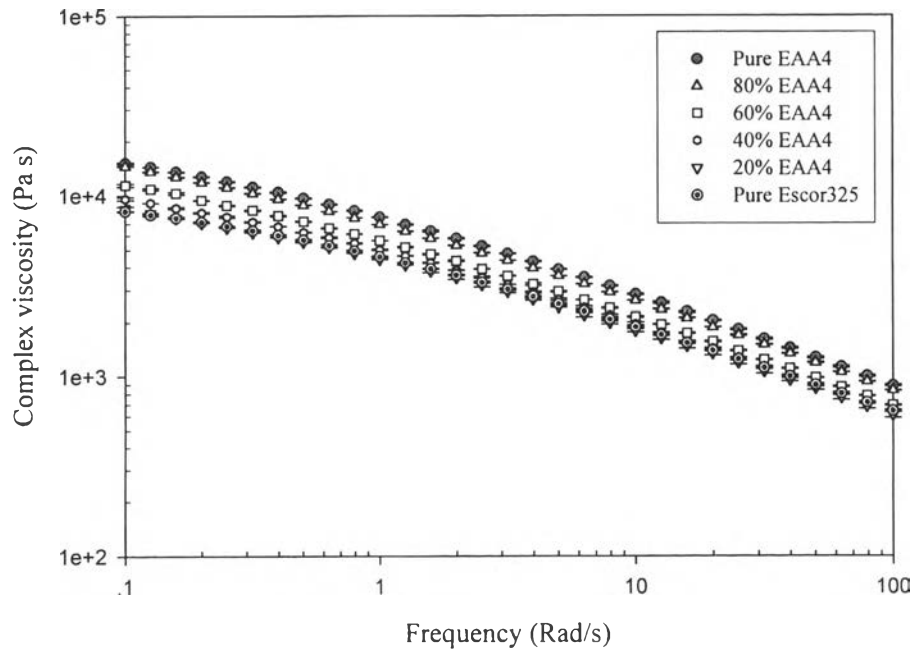


(a)

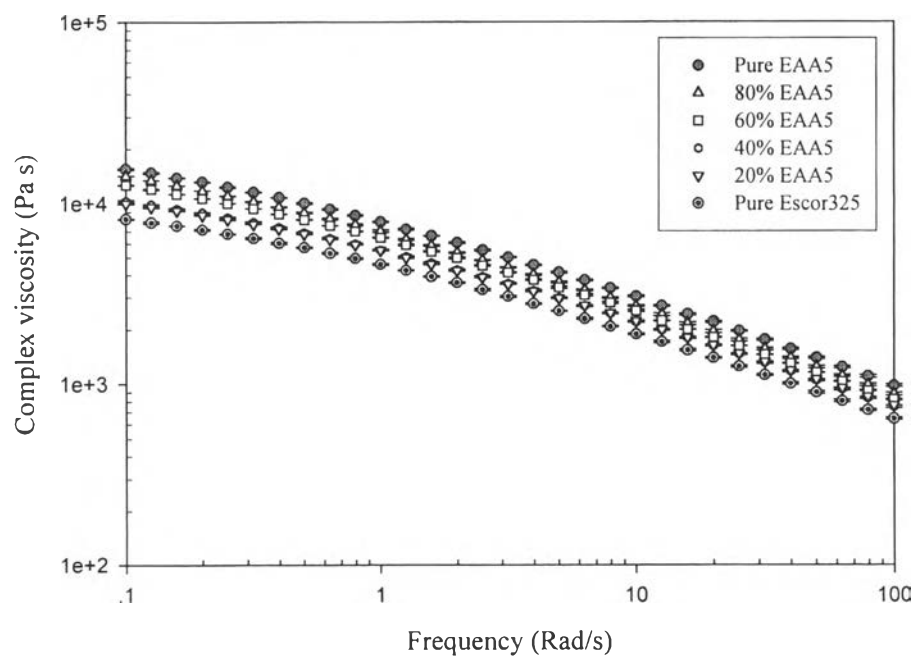


(b)

**Figure1**

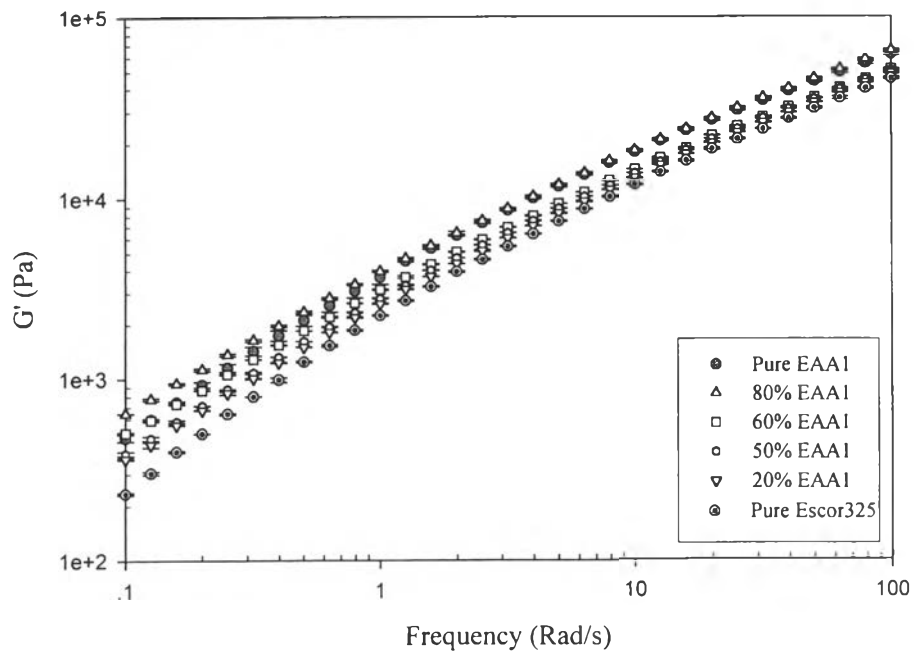


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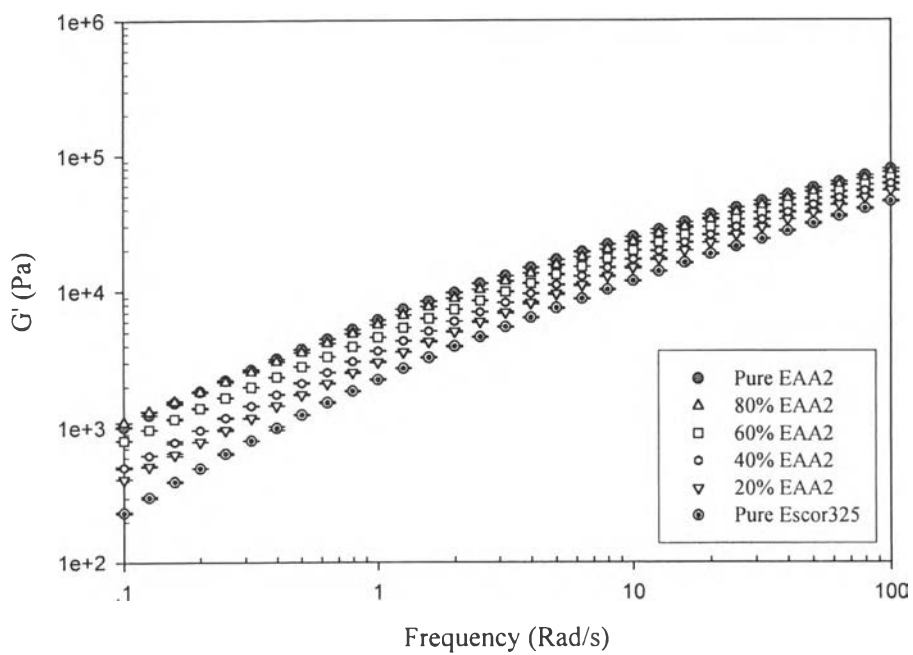


(d)

**Figure 1**

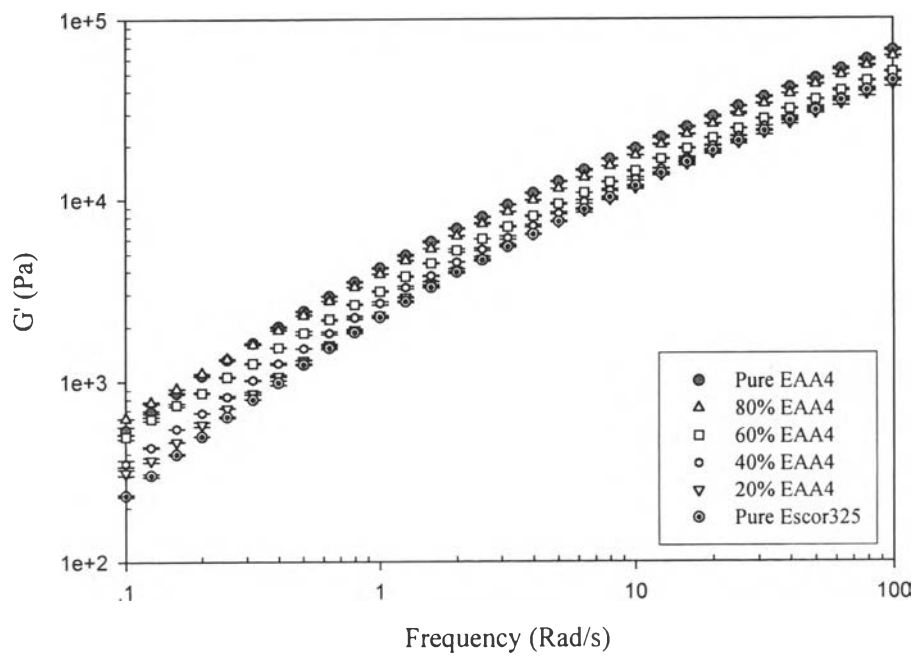


(a)

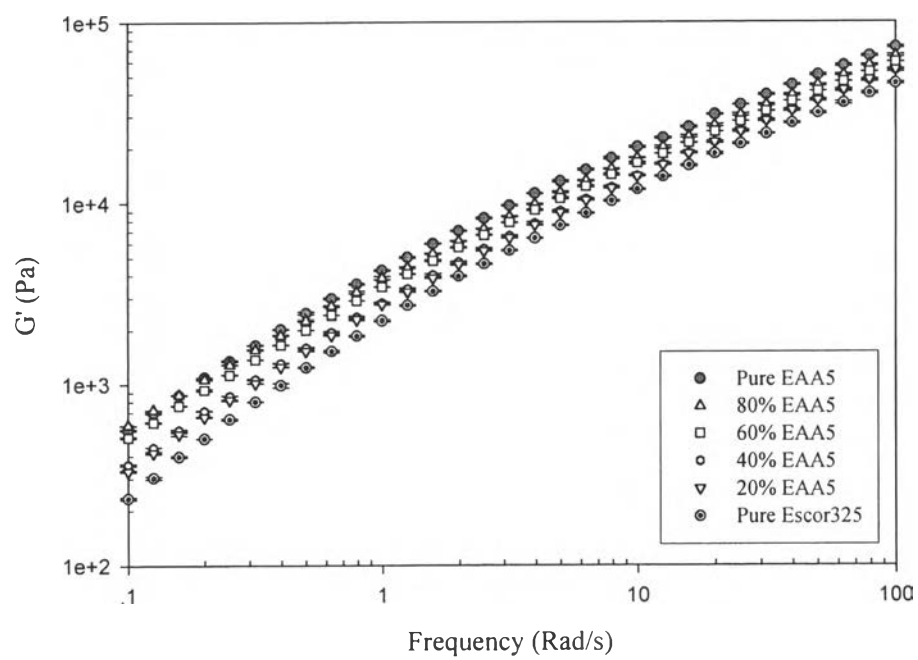


(b)

**Figure2**

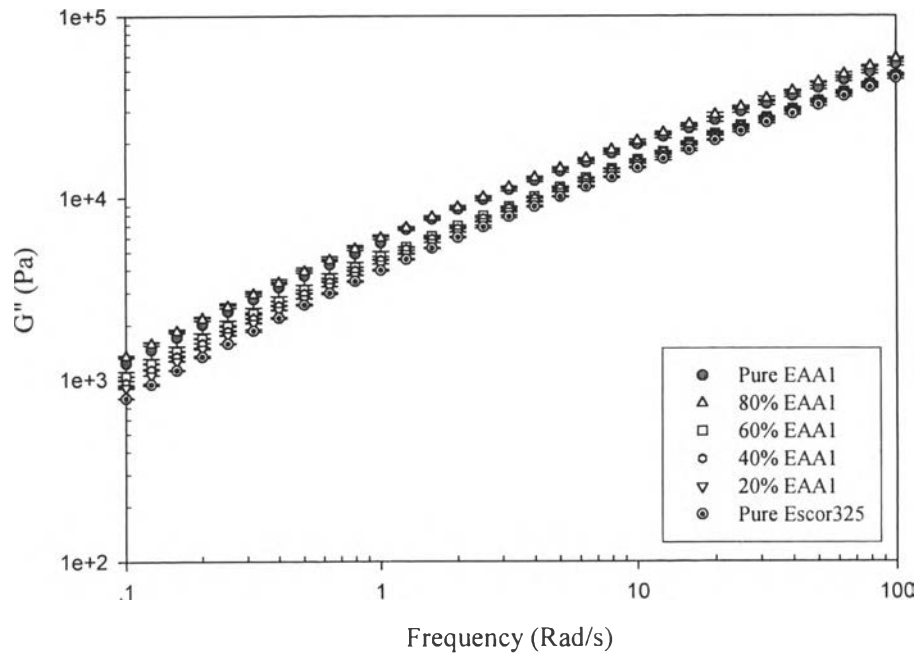


(c)

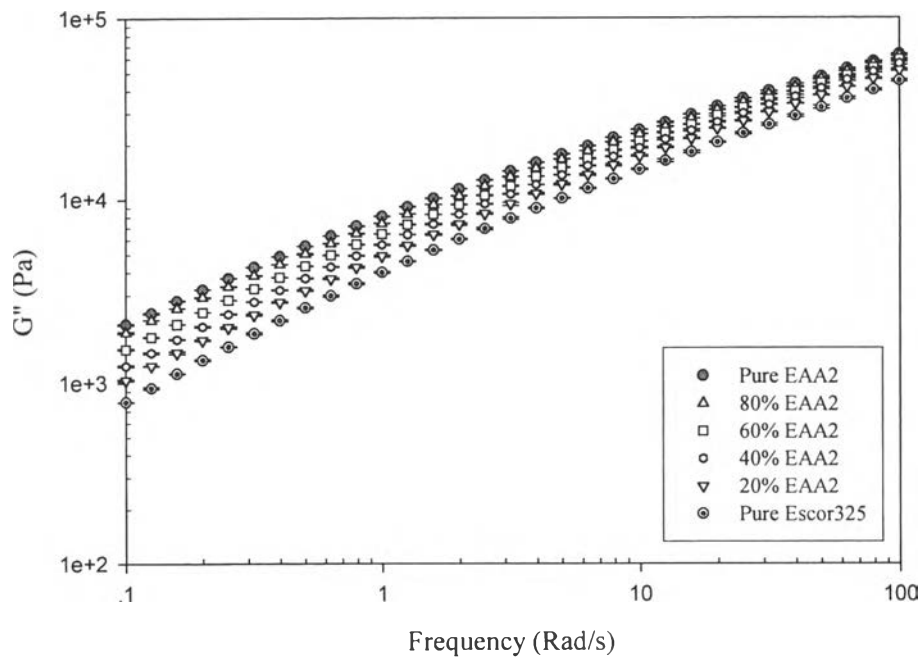


(d)

**Figure 2**

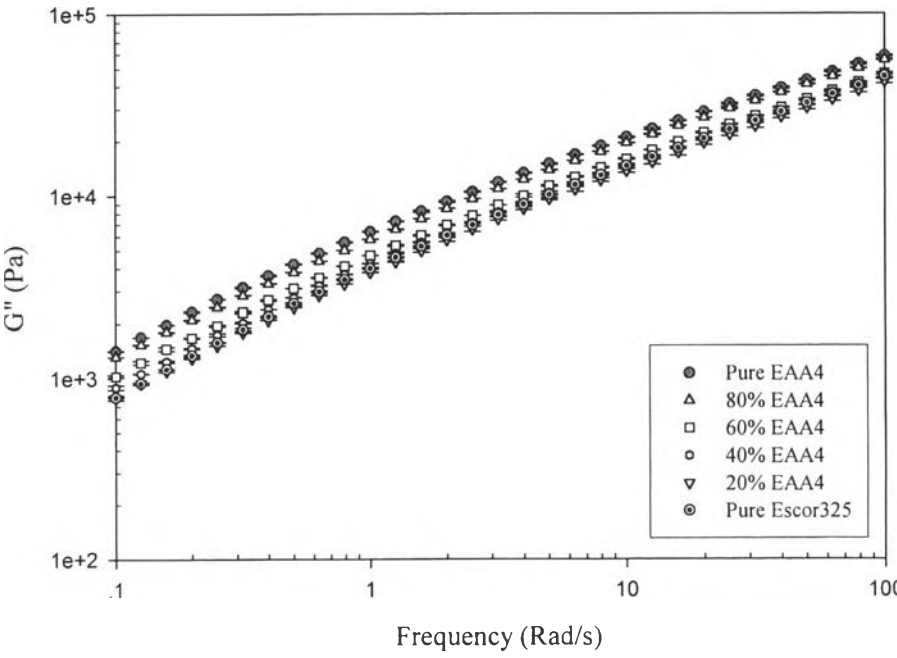


(a)

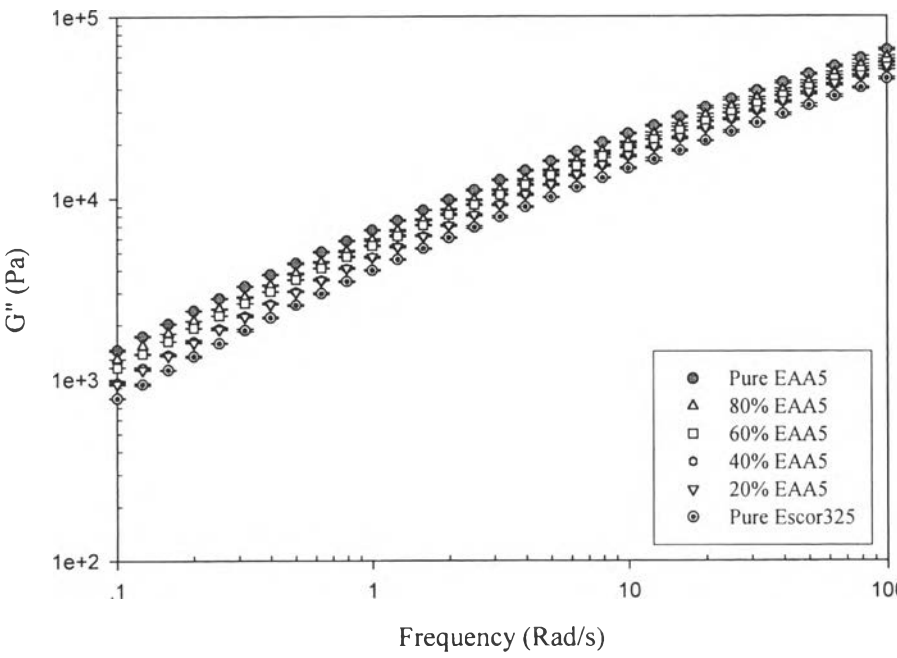


(b)

**Figure 3**

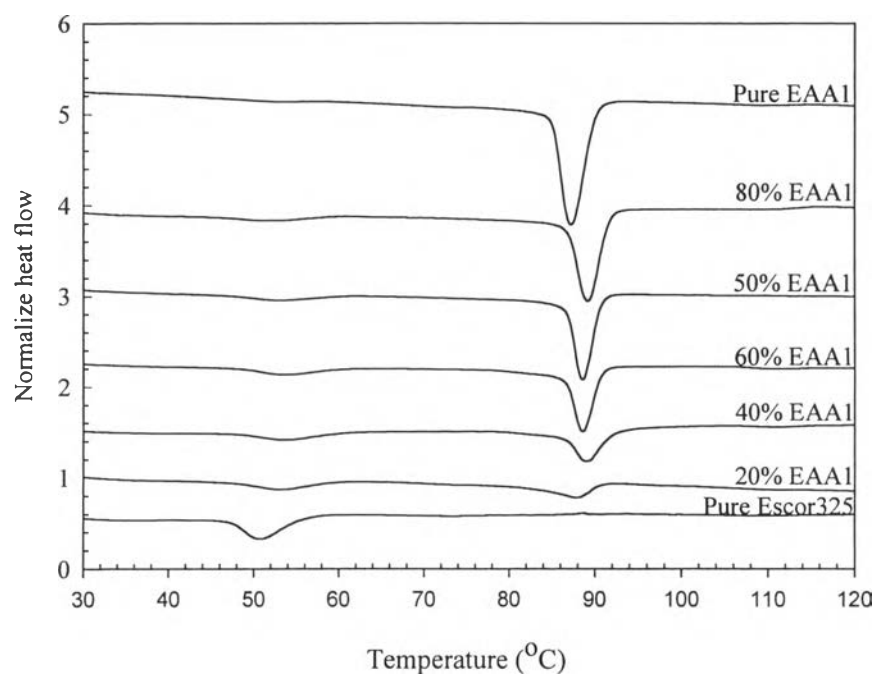


(c)

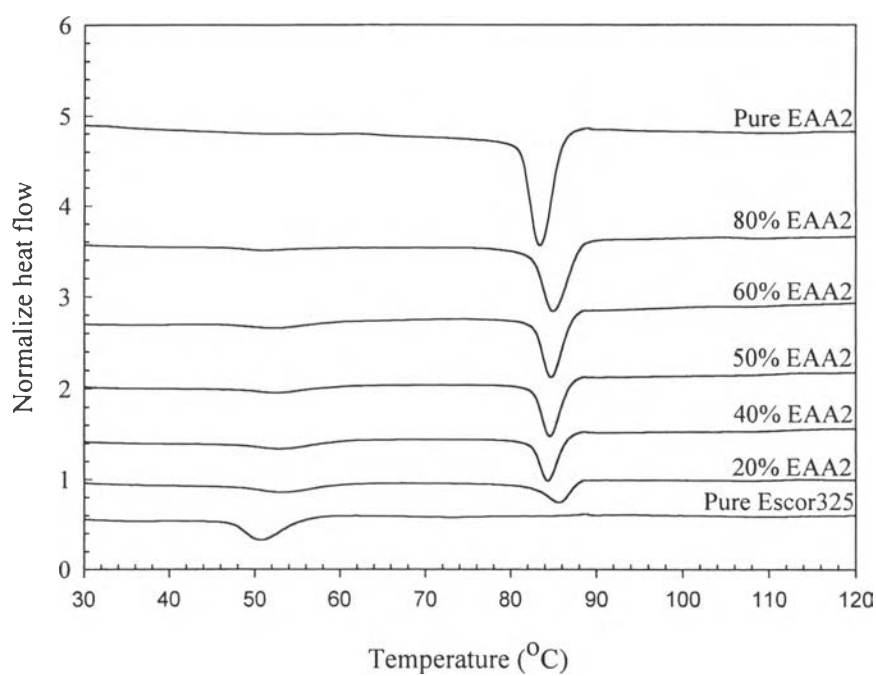


(d)

Figure 3

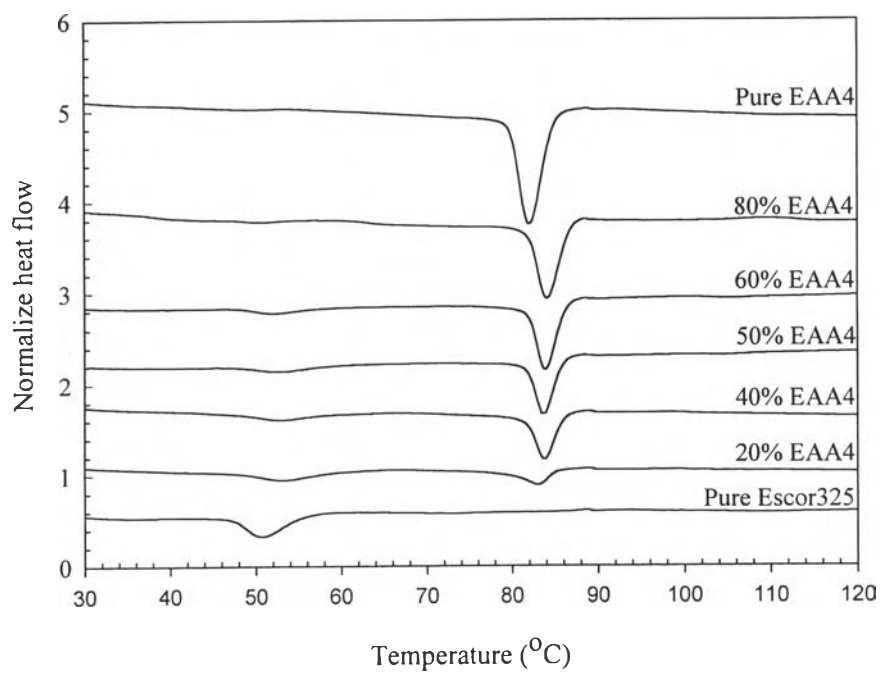


(a)

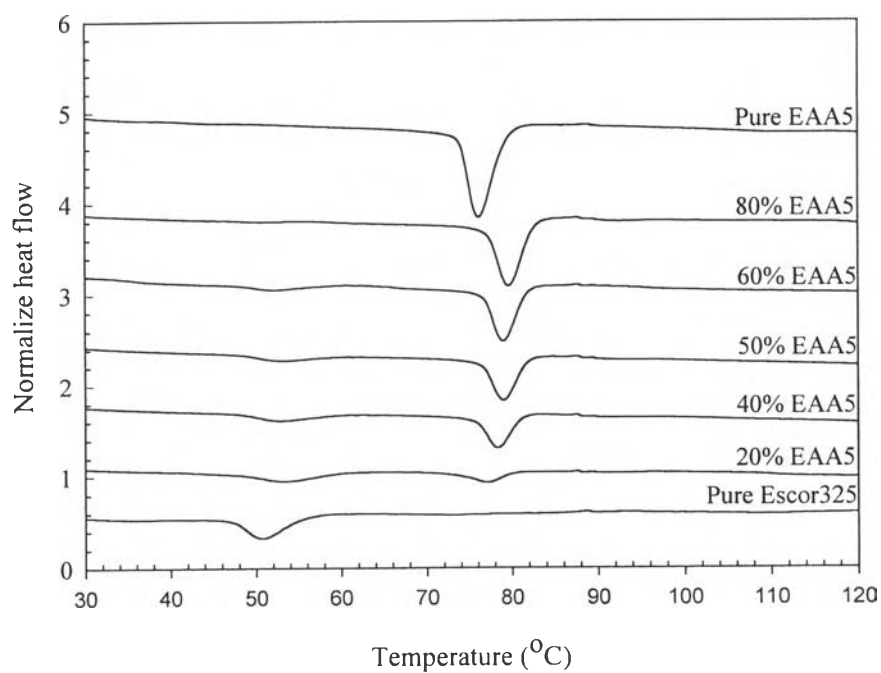


(b)

**Figure 4**



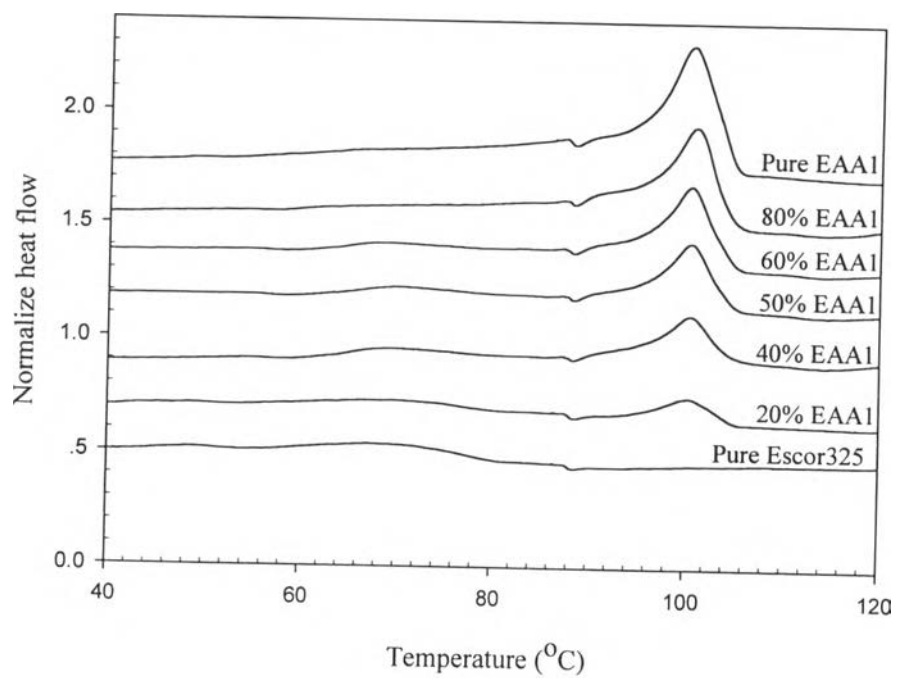
(c)



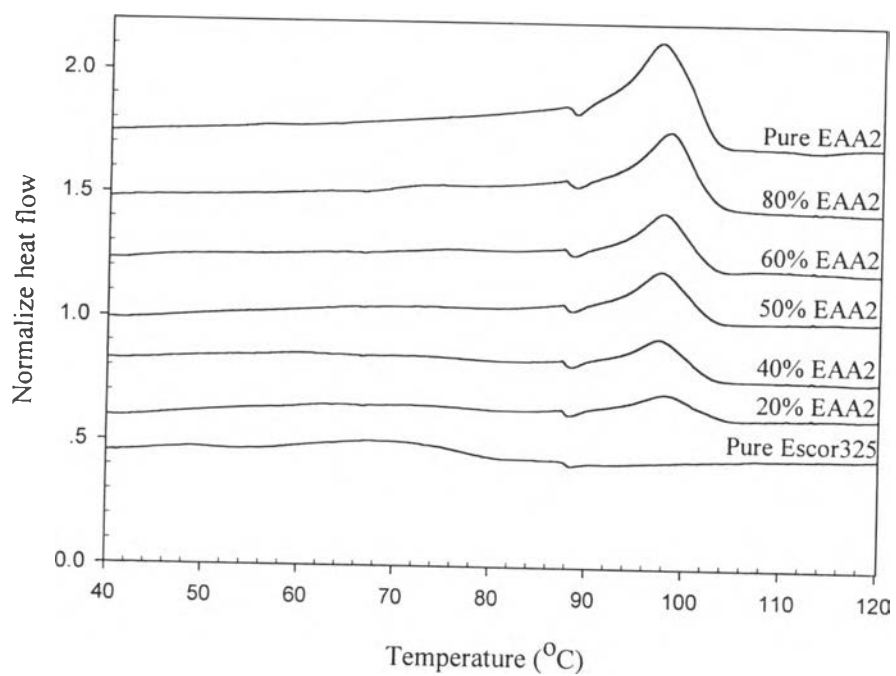
(d)

**Figure 4**



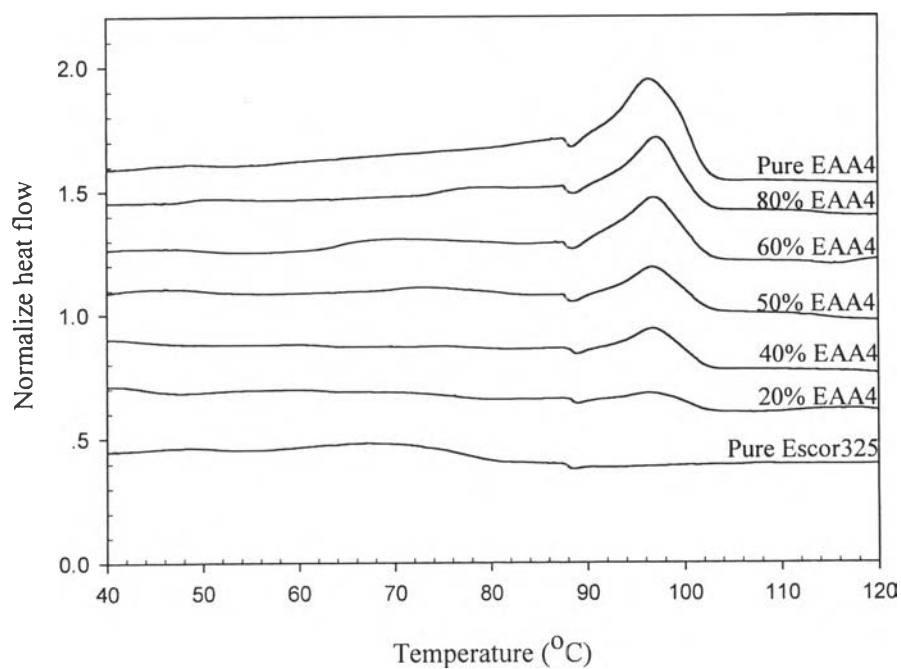


(a)

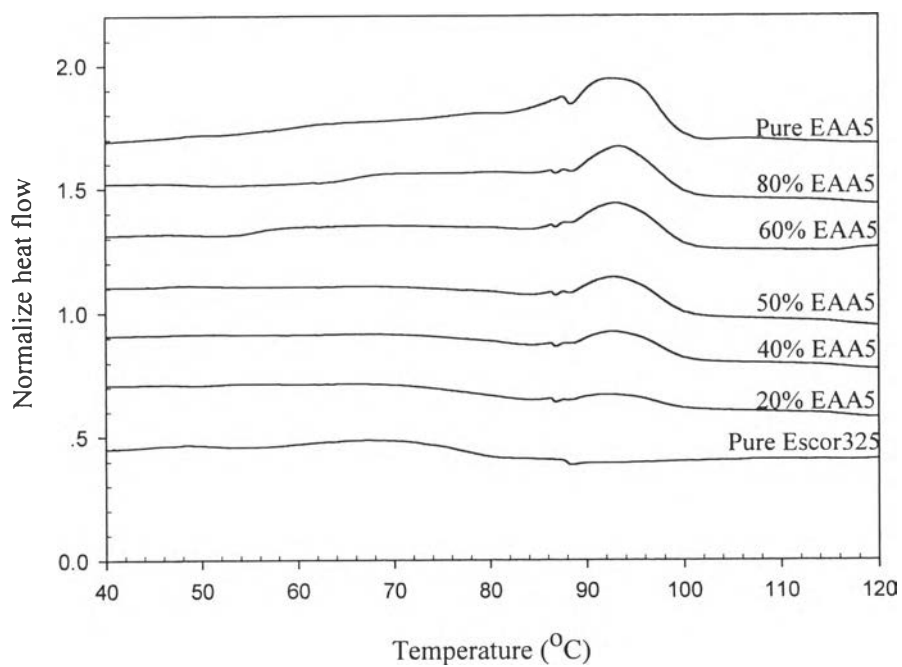


(b)

**Figure 5**

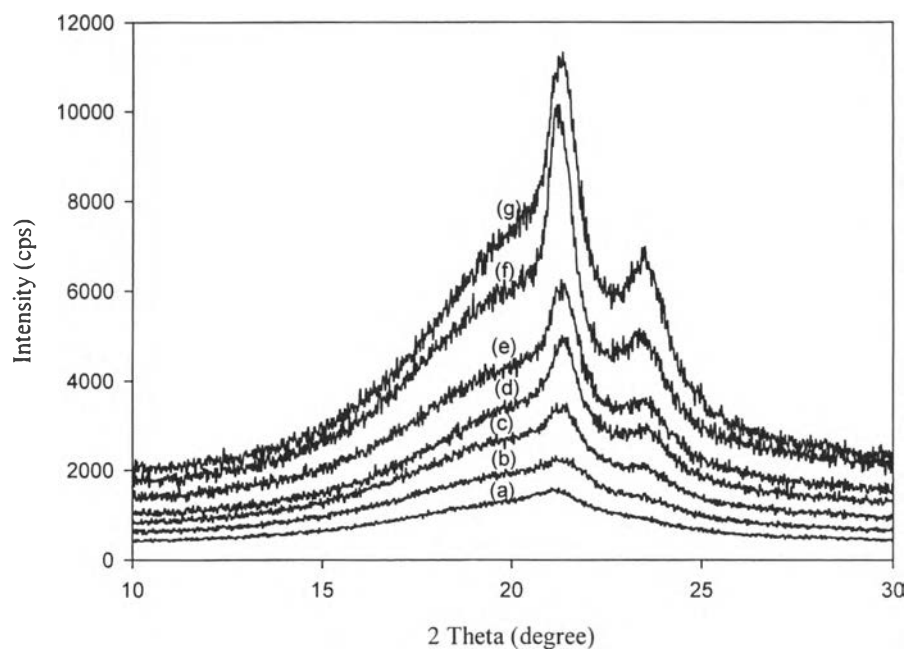


(c)

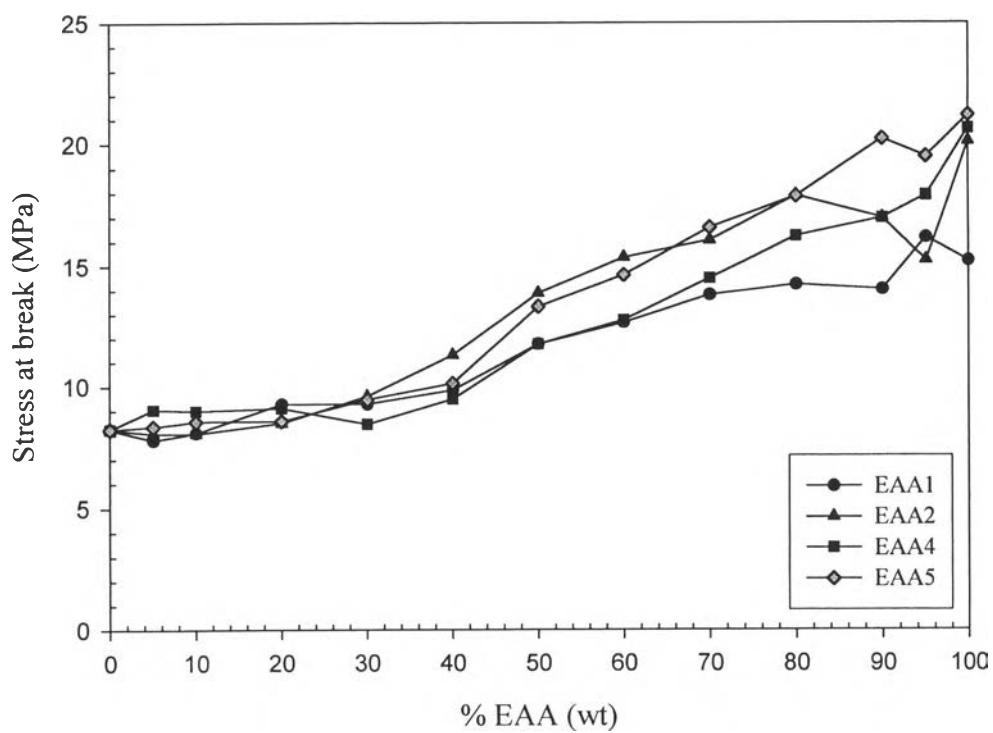


(d)

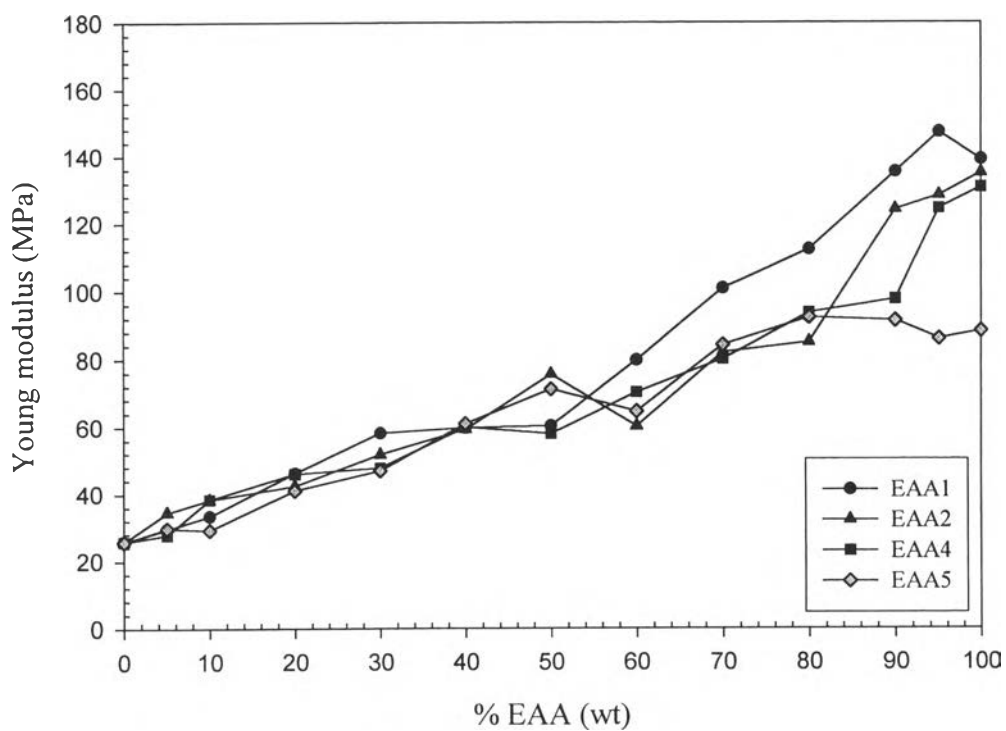
**Figure 5**



**Figure 6**

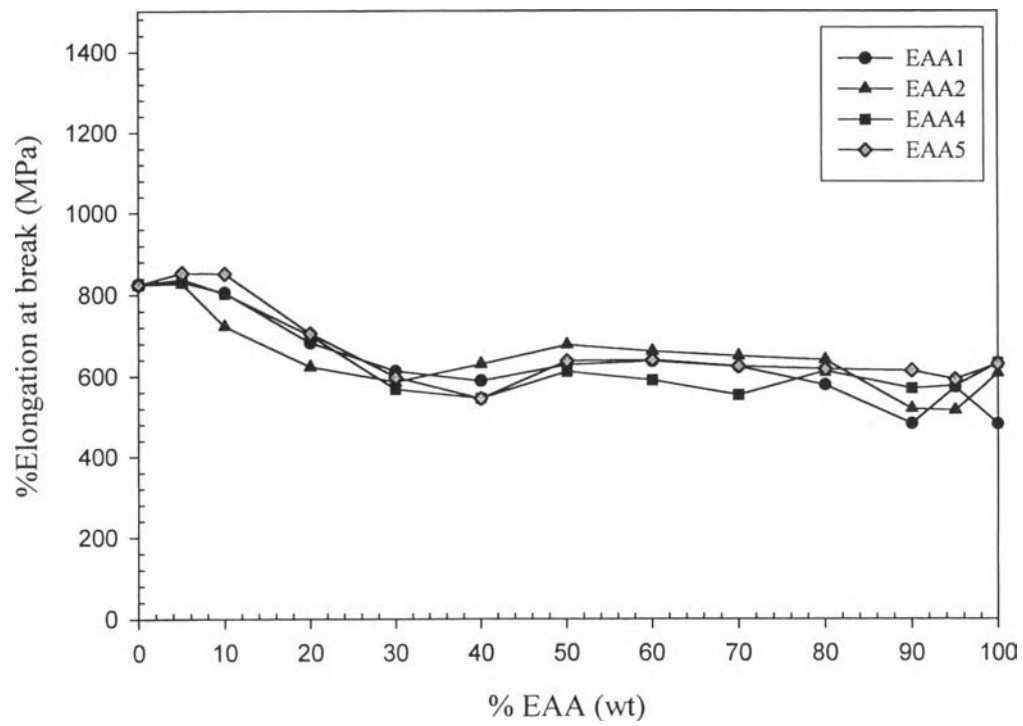


(a)



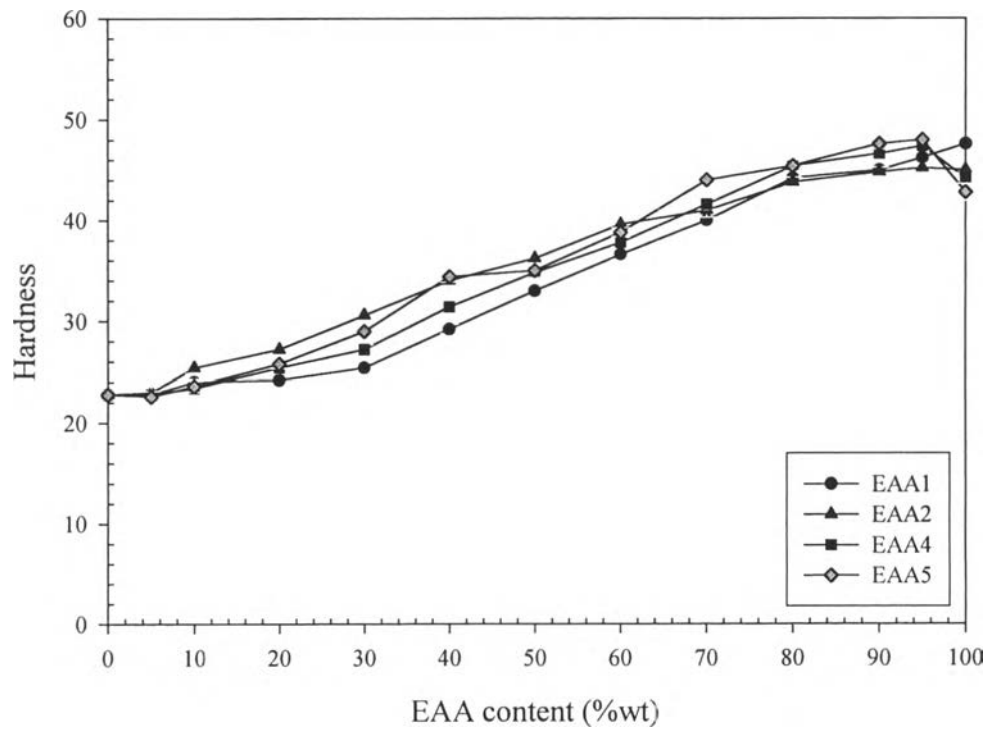
(b)

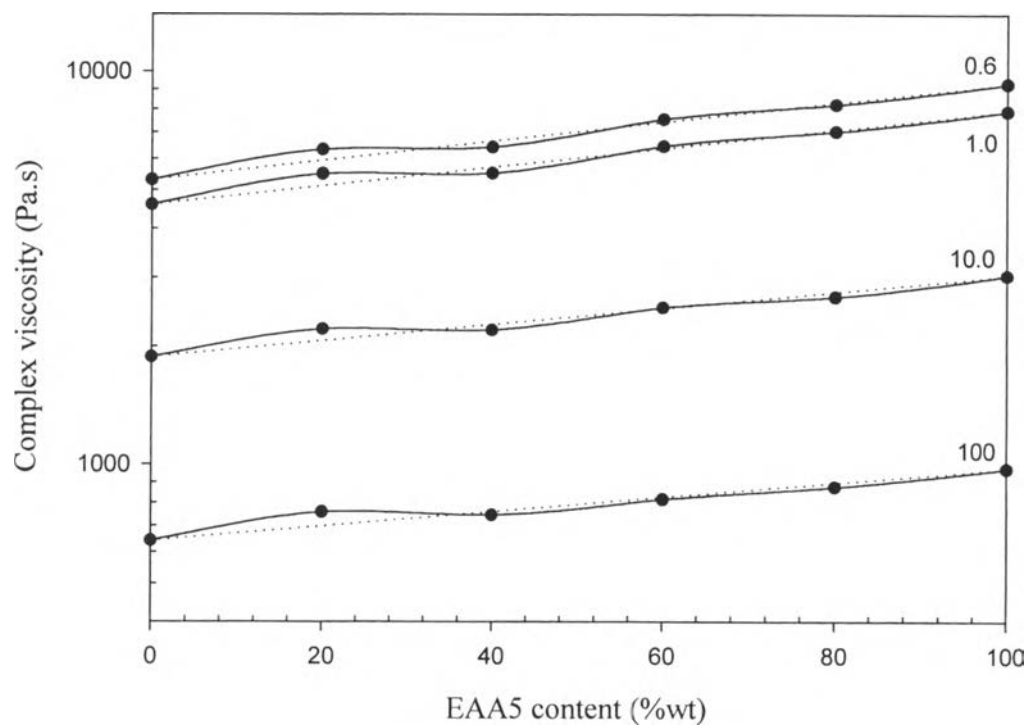
**Figure 7**



(c)

**Figure 7**

**Figure 8**

**Figure 9**