

Easy Preparation of Crosslinked Polymer Films from Polyoxyalkylene Diamine and Poly(styrene–maleic anhydride) for Electrostatic Dissipation

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ABSTRACT: The curing reaction of polyoxyalkylene diamine and poly(styrene–maleic anhydride) was able to produce polymer films that were hygroscopic in nature. The process involved the formation of an imine intermediate by dissolving the diamine monomers in a ketone solvent, followed by instant casting into films before the solution self-cured into a gel product. Hydrophilic films were formed by the fast reaction of amine with anhydride while evaporating the solvent under vacuum. The resulting films exhibited dissipation of electrostatic charges over a wide range, from $10^{9.4}$ to $10^{4.1}$ Ω /sq surface resistivity. The low resistivity was attributed to the

functionality of hydrophilic polyoxyethylene (POE), which was able to adsorb moisture and optionally to the added metal ion salts. Furthermore, the slightly crosslinked network prevented POE from having crystallinity and rendered the films solvent resistant, thermally stable, and suitable for applications as antistatic and polymeric electrolytes. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 103: 716–723, 2007

Key words: films; crosslinking; gelation; polyamines; polyethers

INTRODUCTION

Most hydrophobic polymers used in ordinary applications are generally good electrical insulators, with surface resistivity in the range of 10^{15} – 10^{17} Ω /sq.¹ In electronic packaging and encapsulation applications, plastic materials can easily accumulate electrostatic charges that can cause such side effects as attracting dust or damaging internal electronic components.² Commonly, an organic surfactant or carbon black is blended in to a material during fabrication^{3,4} in order to increase the capacity to dissipate electrostatic charges. Some classes of hydrophilic functional polymers also have low surface resistivity and are already able to dissipate electrostatic charges. However, these polymers are generally too hydrophilic to be compatible with hydrophobic polymers or lack long-term performance. Recent studies indicated that copolymers with polyoxyethylene segments are those most suitable for electrostatic-dissipating applications and as solid electrolytes by chelating with metal-ion conduction.^{5,6}

Copolymers composed of polyoxyethylene (POE) blocks are known to be able to absorb moisture and chelate metal ions.^{7–9} The introduction of a POE block

to a copolymer structure may reduce its surface resistivity in the range of 10^8 – 10^{10} Ω /sq¹⁰ or even lower, to 10^4 – 10^6 Ω /sq¹¹, when associating with metal ions. This surface resistivity is significantly lower than that of a hydrophobic polymer ($> 10^{12}$ Ω /sq). Previous studies also found that lowering surface resistivity may be limited by the inherent crystallinity of POE,¹⁰ which restricts polymer chain mobility and hence ionic conductivity. Maintaining polymer flexibility while increasing the weight fraction of POE and solvated metal ions subsequently affects the efficiency of a polymer's electrostatic dissipation and ionic conduction. Hence, a polymer that is amorphous or has low crystallinity is more suitable than a crystalline material for applications as antistatic and solid electrolyte materials.

Previously, we reported the uses of a polyoxyethylene diamine with a molecular weight of 2000 g/mol as a POE building block for constructing POE-segmented copolymers with moderate crystallinity.¹² We also developed crosslinkable macromonomers containing cyanuric chloride as the linking functionality for the preparation of POE-containing polymer films that greatly dissipate electrostatic charges ($< 10^5$ Ω /sq).¹¹ Trying to further minimize copolymer crystallinity and improve the film's properties prompted us to develop a new method for preparing POE-segmented and crosslinked networks. In this article, we report an easy synthetic method for producing polymer films involving the reaction of highly reactive

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anhydride and amine functionalities. The *in situ* polymerization of poly(styrene-maleic anhydride) and POE diamine could afford polymeric films with a POE network of low crystallinity. The electronic conductivity, thermal stability, and ionic chelation properties were evaluated and correlated with the POE structures.

EXPERIMENTAL

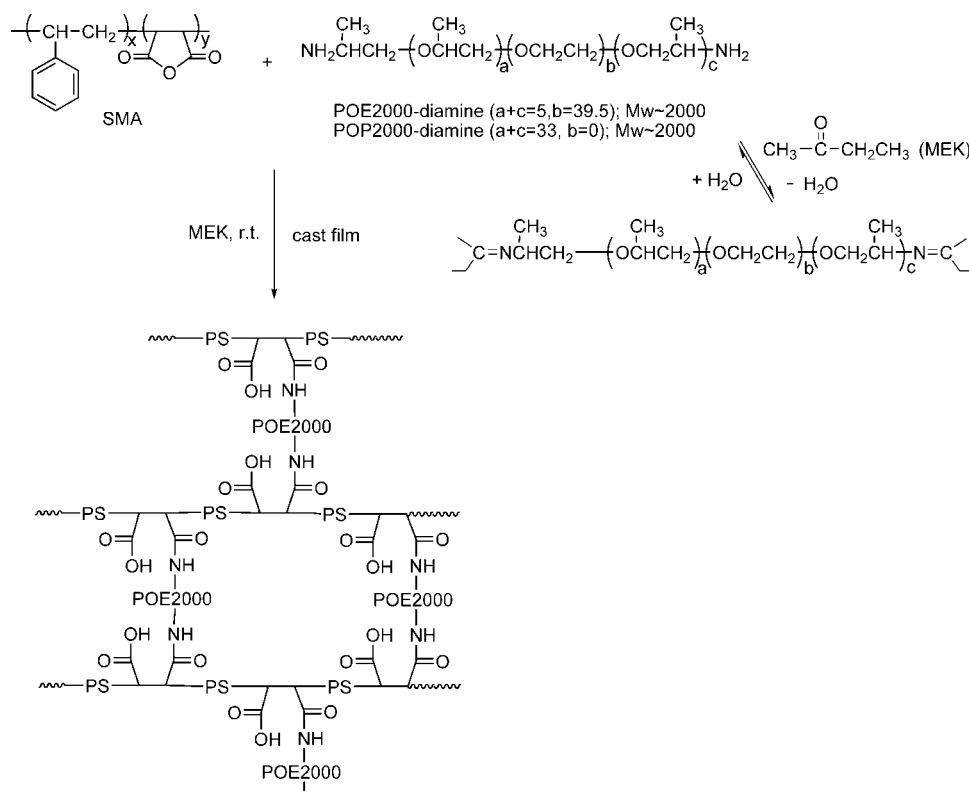
Materials

Poly(styrene-maleic anhydride) copolymer with a weight-average molecular weight of 90,700, a number-average molecular weight of 34,200 g/mol, and a maleic anhydride (MA) content of approximately 14 wt % was obtained from the Aldrich Chemical Co. (Milwaukee, WI). The molar ratio of styrene to MA was estimated to be 6 : 1. The molecular weight was confirmed by gel permeation chromatography (GPC) analysis. Polyoxyalkylene diamines, including amines with polyoxyethylene (POE) and polyoxypropylene (POP) backbones, are commercially available under the trade name Jeffamine[®] and were purchased from Huntsman Chemical Co. or Aldrich Chemical Co. POE2000 diamine (white waxy solid, m.p. 35°C, amine content 0.95 mequiv/g, $M_w \sim 2000$) has a chemical structure consisting of a middle POE block and two short

oxypropylene repeating end blocks with an average oxyethylene/oxypropylene unit ratio of 39.5 : 5, as shown in Scheme 1. The POE2000 diamine was dried in an oven under vacuum at 110°C for 3 h before use. POP2000 is a polyoxypropylene-backboned diamine with a weight-average molecular weight of approximately 2000 and an amine content of 0.98 mequiv/g. Oxypropylene-backboned POP2000 is a water-insoluble diamine; in contrast, oxyethylene-rich POE2000 is hydrophilic and water soluble. An amine modifier, *N,N*-dimethyl aminopropylamine (DAP), was obtained from the Aldrich Chemical Co. The metal salts including magnesium chloride, lithium chloride, lithium perchlorate, nickel chloride, and copper chloride were purchased from Showa Chemical Co. and dried under reduced pressure before using.

Measurements

Surface resistivity was measured with a model ST-3 tester (Simco Co.) according to ASTM method D257-93. Measurement was in units of Ω/sq , in which the size of the square is immaterial. All samples were conditioned in an atmosphere of 50% relative humidity before measurement. Samples were dissolved in methyl ethyl ketone (MEK), evaporated into thin films on a KBr plate, and analyzed using an FTIR spectrometer (Perkin Elmer Spectrum One FTIR spectrometer).



Scheme 1 Synthetic scheme illustrating polymer film formation from SMA-POE2000 and SMA-POP2000 at ambient temperature.

TABLE I
Relative Gel Time for Reaction of SMA (14 wt % MA)
and POE2000 Diamine^a

Solvent	Weight fraction (wt %) ^b	Gel time (s)
Toluene	25	50
Toluene	20	80
Toluene	17	120
MEK	25	315
MEK	20	505
MEK	17	710

^a Reactions were performed with a controlled stirring rate and identical masses.

^b Weight fraction (wt %): solid content of SMA + POE2000 (at molar ratios of MA/POE2000 of 1 : 0.5 and MA/NH₂ of 1 : 1) in solvent.

Thermal gravimetric analysis (TGA) was performed on a Perkin Elmer Pyris 1 by heating a sample from room temperature to 550°C at a rate of 10°C/min in nitrogen. Analyses of crystalline temperature and enthalpy were carried out with a differential scanning calorimeter (DSC; TA DSC 2010). A 3- to 8-μg sample in a sealed aluminum pan was generally used. For the scanning, the temperature was increased from -20°C from 180°C at a heating rate of 10°C/min with a nitrogen flow of 20 mL/min.

Preparation of SMA-POE2000 polymer films

Typical procedures for preparing an instant SMA-POE2000 crosslinked film are described below. A round-bottomed flask equipped with a magnetic stirrer was charged with the POE2000 diamine (2.77 g, 1.39 mmol) and MEK (6 mL), followed by the addition

of a solution of the SMA copolymer (2.00 g, 2.77 mmol of MA) dissolved in 6 mL of MEK. The solution was mechanically stirred for 30 s and immediately coated on a glass slide. A thin film was obtained after evaporating the solvent at ambient temperature under vacuum for 2 h. The reaction was monitored by FTIR spectroscopy for the peak absorptions, 1662, 1711, 1580, and 1105 cm⁻¹, corresponding to symmetric stretch of the C=O of amide, symmetric stretch of the C=O of acid, stretch of the C-N in amide, and the C-O-C in the ether group, respectively.

Preparation of metal-ion-doped SMA-POE2000 polymer film

As an example, SMA-POE2000-NiCl₂ film is described for a molar ratio of Ni⁺/O at 1 : 10 on the basis of the oxyethylene unit in the POE2000 segment. To a round-bottomed flask equipped with a magnetic stirrer, nickel chloride (0.80 g; 6.17 mmol) in ethanol (5 mL) and POE2000 diamine (2.77 g; 1.39 mmol) in MEK (4 mL) were charged while stirring. Then, SMA (2.00 g, with the calculated 2.77 mmol of MA) in MEK (5 mL) was added and vigorously stirred for 30 s. A thin film was prepared by quickly coating the homogeneous mixtures on a glass slide and subjecting them to evaporation at ambient temperature for 12 h and in a vacuum oven for 2 h before analysis.

RESULTS AND DISCUSSION

Preparation of polymer films with crosslinked POE segments

Previously, we developed a method for grafting POE-amines onto maleated polystyrene-poly(ethylene/

TABLE II
Surface Resistivity of Polymer Films with Different POE and DAP Fractions

Film composition	MA/POE2000/DAP (molar ratio) ^b	POE weight fraction (wt %)	Surface resistivity (10 ^x Ω/sq)
SMA	1 : 0 : 0	0	> 12
POE2000 diamine	0 : 1 : 0	100	10
SMA-POE2000	1 : 0.25 : 0	41	9.4
SMA-POE2000 ^a	1 : 0.5 : 0	58	8.6
SMA-POE2000	1 : 0.75 : 0	68	8.2
SMA-POE2000	1 : 1 : 0	74	6.8
SMA-POP2000 ^c	1 : 0.5 : 0	58	> 12 ^e
SMA-POE2000-DAP	1 : 0.25 : 0.5	39	9.7
SMA-POE2000/DAP-H ⁺ (1 : 0.5) ^d	1 : 0.25 : 0.5	39	6.1
SMA-POE2000/DAP-H ⁺ (1 : 1)	1 : 0.25 : 0.5	39	5.2

^a MA/NH₂ ratio of 1 : 1 in SMA-POE2000 crosslinking films.

^b DAP, *N,N*-dimethylaminopropylamine H₂NCH₂CH₂CH₂N(CH₃)₂.

^c POP2000, a diamine with a polyoxyethylene backbone of 2000 g/mol compared with POE2000.

^d Acidification by adding HCl.

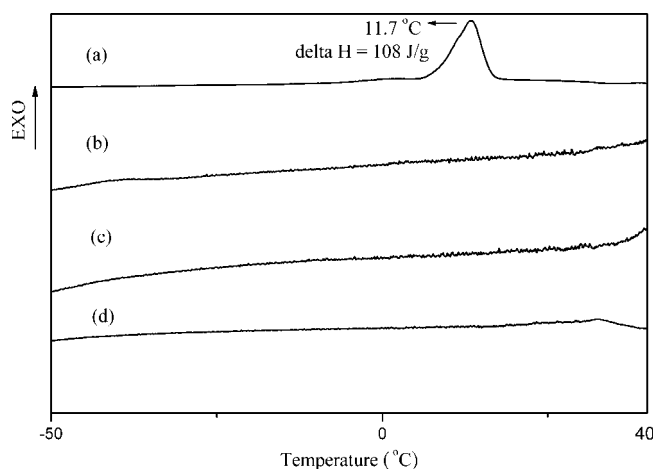


Figure 1 DSC curves of starting material: (a) POE2000 diamine and prepared films, (b) SMA-POE2000 at MA/NH₂ = 1 : 2, (c) SMA-POE2000 at MA/NH₂ = 1 : 1.5, (d) SMA-POE2000 at MA/NH₂ = 1 : 1.

butylene)-polystyrene (SEBS-*g*-MA)¹³ without forming a gel product. The amine functionality of the hydrophilic POE-monoamine and diamines were readily grafted onto the reactive moieties of succinic anhydride in the SEBS-*g*-MA polymer backbone. Because of the low anhydride content of SEBS-*g*-MA (2 wt %), it was possible to avoid the crosslinking reaction of the POE diamines with SEBS-*g*-MA, and a nongelled copolymer was prepared. However, a polymer gel was actually observed in attempting to graft the amines onto a poly(styrene-maleic anhydride) (SMA) copolymer with an anhydride content of more than 14 wt %. A crosslinking reaction between the SMA and POE diamine to form gels could only be postponed by using a specific solvent. Under an identical stirring rate, the reaction of SMA with POE2000 diamine in toluene (25 wt %) produced a gel within 50 s, but in MEK as the solvent it took 315 s (Table I). A systematic study indicated that gel formation was much slower in a ketone solvent such as MEK than in toluene, perhaps because of the formation of an equilibrium imine^{14,15} intermediate (C=N-) between the POE2000 amine and the ketone solvent. By adjusting the amount of MEK solvent, the gel time could be prolonged from 50 to 710 s. This finding allows us to develop a convenient method for preparing polymer films. Scheme 1 is a conceptual diagram representing a polymer network of POE2000 segments in linking polystyrene through multiple amido acid sites. By taking advantage of the solvent effect, thin films could be prepared by simply casting or spin-coating the homogeneous solutions within the gel time, followed by removal of the solvent through evaporation. Heating was optionally applied to increase the amidation rate, although the films could be formed readily at room temperature by slowly removing the solvent.

Effect of crosslinking structure on ionic conduction

Polymerization of SMA and POE diamine at a fast rate could lead to the convenient preparation of polymer films with enhanced conductivity. According to the ionic hopping mechanism,^{16,17} the efficiency of polymer electronic conducting is a function of its ionic charge mobility. It is known that the presence of POE segments in copolymers may contribute to both hygroscopic and metal-ion chelating abilities but may adversely affect polymer flexibility. For compromising the effects, a suitable structure is optimized by increasing the POE component but mitigating its inherent crystallinity. By controlling the crosslinking reaction between SMA and diamine, polymer films with low crystallinity can be prepared. As summarized in Table II, the films derived from SMA and POE2000 generally exhibited low surface resistivity, in the range of 10^{9.4}-10^{6.8} Ω/sq. The trend for electrostatic dissipating ability could be correlated with the increased POE weight fraction. The lowering of surface resistivity was a function of the increase in the POE fraction from 41 to 74 wt %. Because the POE2000 starting material itself in bulk has high resistivity (10¹⁰ Ω/sq), the crystallinity is attributed to the electronic conductivity in adversity. An examination of their thermal properties with differential scanning calorimetry showed POE2000 had high crystallinity, with a crystalline temperature (*T_c*) of 11.7°C (Figure 1). However, the prepared SMA-POE films exhibited no

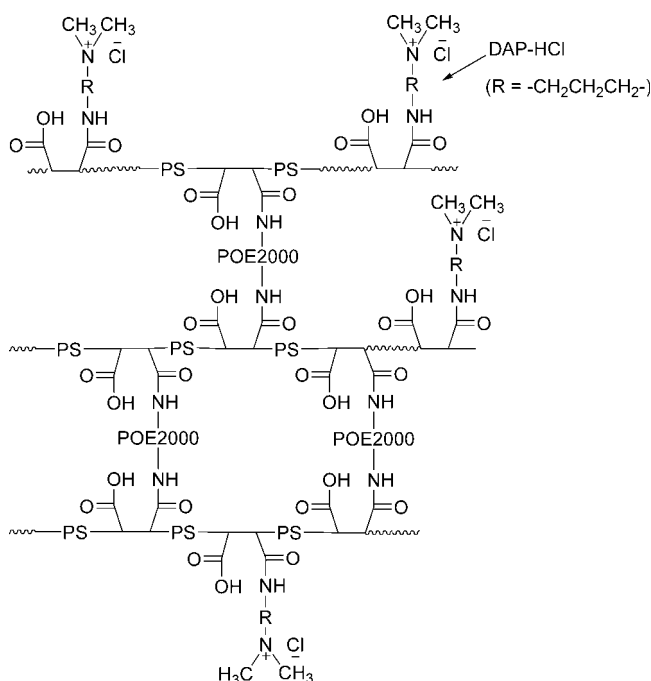


Figure 2 Idealized chemical structure of SMA-POE2000/DAP-H⁺ cationic films prepared by the addition of DAP and acidification.

TABLE III
Surface Resistivity of Polymer Films with Various Amounts of Metal Ion Doping

Entry	SMA-POE2000/ metal	POE2000 fraction (wt %) ^c	O/M ⁿ⁺	Surface resistivity (10 × Ω/sq)
1	None ^a	58	0	8.6
2	LiClO ₄ ^a	57.8	300	7.6
3	LiClO ₄ ^a	57.7	200	6.6
4	LiClO ₄ ^a	57.3	100	6.4
5	LiClO ₄ ^a	51.0	10	5.4
6	LiClO ₄ ^a	45.5	5	4.3
7	None ^b	74.0	0	6.8
8	LiClO ₄ ^b	73.0	300	6.7
9	LiClO ₄ ^b	72.8	200	6.4
10	LiClO ₄ ^b	72.2	100	5.9
11	LiClO ₄ ^b	62.5	10	4.9
12	LiClO ₄ ^b	54.4	5	4.1
13	LiCl ^a	58.0	300	7.3
14	LiCl ^a	57.9	200	6.7
15	LiCl ^a	57.8	100	6.4
16	LiCl ^a	55.1	10	4.7
17	LiCl ^a	52.4	5	4.6
18	NiCl ₂ ^a	57.7	300	7.4
19	NiCl ₂ ^a	57.6	200	6.8
20	NiCl ₂ ^a	57.1	100	6.2
21	NiCl ₂ ^a	49.7	10	4.8
22	NiCl ₂ ^a	43.5	5	4.2
23	CuCl ₂ ^a	57.7	300	7.1
24	CuCl ₂ ^a	57.5	200	6.0
25	CuCl ₂ ^a	56.9	100	6.0
26	CuCl ₂ ^a	47.9	10	4.8
27	CuCl ₂ ^a	40.8	5	4.3
28	MgCl ₂ ^a	57.8	300	7.3
29	MgCl ₂ ^a	57.7	200	6.9
30	MgCl ₂ ^a	57.4	100	6.5
31	MgCl ₂ ^a	51.7	10	4.9
32	MgCl ₂ ^a	46.6	5	4.3

^a Molar ratio of MA/POE2000 is 1 : 0.5 (MA/NH₂ = 1 : 1).

^b Molar ratio of MA/POE2000 is 1 : 1 (MA/NH₂ = 1 : 2).

^c Weight fraction (wt %): POE2000/SMA+POE2000+metal species.

^d No apparent change in surface resistivity as O/Mⁿ⁺ < 5.

obvious T_c because of the presence of a crosslinking structure, which did not allow POE aggregation. Hence, in slightly crosslinking the hydrophilic POE segment was advantageous for the electrostatic dissipating behavior. In comparison, the hydrophobic POP2000-amine could only produce SMA films with low electrostatic dissipation ($> 10^{12} \Omega/\text{sq}$). It appears that the hydrophilic POE segment was different from POP2000 in adsorbing moisture.¹² Enhancement of electronic conductivity is dictated by the hydrophilic behavior of POE segments involving the mechanism of hydrogen-bonding association between $-(\text{CH}_2\text{CH}_2\text{O})_x-\text{H}_2\text{O}$ and possible water ionization.¹¹ For the SMA-POE2000 films, the slight crosslinking network reduced the negative effect of POE crystallinity and consequently improved electrostatic dissipation.

The structure of the SMA-POE2000 films could be further modified by grafting short amine pendants in the comb-branched copolymers. The amine pendants

could provide reactive sites for generating ionic charges upon acidification. The use of *N,N*-dimethylamino-propyl amine (DAP) for partial grafting on the SMA

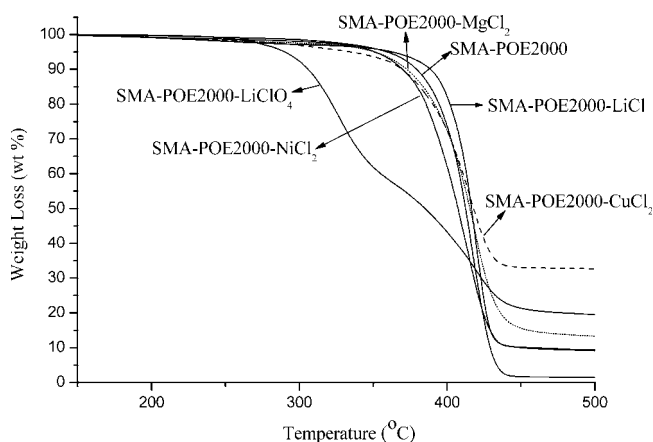


Figure 3 TGA of SMA-POE2000 films with different metal salts at a molar ratio of O/Mⁿ⁺ = 10.

TABLE IV
TGA Decomposition Patterns of Metal-Ion-Doped SMA-POE2000 Film in Nitrogen

	Temperature (°C) at				Char yield (%) at	
	95%	75%	50%	25%	450°C	500°C
	Retention (wt %)					
SMA-POE2000	359	399	412	422	0	0
SMA-POE2000-LiCl ^a	363	405	416	424	10	9
SMA-POE2000-LiClO ₄ ^a	290	329	385	432	21	19
SMA-POE2000-MgCl ₂ ^a	351	397	415	431	16	14
SMA-POE2000-CuCl ₂ ^a	340	397	418	— ^b	33	32
SMA-POE2000-NiCl ₂ ^a	350	391	407	421	10	9

^a O/metal ion = 10 : 1.

^b Char yield at 550°C = 33 wt %.

led to a structure with multiple pendent amine functionalities. In combining POE2000 and DAP, the polymer films were tailored with multiple tertiary amines. Figure 2 shows an idealized structure that illustrates the polymer compositions consisting of SMA backbones, crosslinked POE segments, and amine pendants. These DAP-doped films appear to have only moderate surface resistivity at $10^{9.7}$ Ω/sq. With simple HCl treatment to convert amines into their corresponding amine salts, conducting performance was significantly enhanced, to the range of $10^{6.1}$ – $10^{5.2}$ Ω/sq. The improvement in surface resistivity in SMA-POE2000/DAP-H⁺ can be explained by the presence of a quaternary salt ionic charge in the polymer matrix, in which the ionic conductance was largely reinforced.

Metal-ion solvating copolymers

Furthermore, ionic conductance was affected 1–2 orders of magnitude by adding metal salts. The addition of metal ions actually shifted the conducting mechanism from moisture adsorbing to ionic conducting.¹⁸ It is well documented that through metal ion chelating, the POE segments of polymer matrices may enhance electronic conductivity. The effectiveness is a function of the amount of metal-ion-chelating species or the $-(\text{CH}_2\text{CH}_2\text{O})_x-/M^+$ concentration, the ionic species, and also polymer flexibility. Table III shows a comparison of the different metal ions in association with the POE-crosslinked films. SMA-POE2000 films containing a 1 : 0.5 or 1 : 1 ratio of MA/POE demonstrated surface resistivity of $10^{8.6}$ and $10^{6.8}$ Ω/sq, respectively. With the added LiClO₄ increasing from a molar ratio of 300 : 1 to 5 : 1 of oxygen/metal ion or O/Li⁺, the surface resistivity dropped from $10^{7.6}$ to $10^{4.3}$ Ω/sq. In another SMA-POE2000 polymer film at 1 : 1 MA/amine or 70 wt % POE fraction, resistivity as low as $10^{4.1}$ Ω/sq was achieved. These trends of lowering surface resistivity could be generalized by different metal-ion additions including LiCl, NiCl₂, CuCl₂, and

MgCl₂, as summarized in Table III. It is noteworthy that the lowest surface resistivity, $10^{4.1}$ Ω/sq, observed in the SMA-POE system perhaps sets the limitation that the ionic hopping mechanism can be achieved.

Thermal stability

The presence of $(\text{CH}_2\text{CH}_2\text{O})_x-/M^+$ chelating complexes in polymer matrices is required for effective ionic conduction.^{19,20} However, the metal-oxygen interaction tends to destabilize the POE segments because of possible weakening of C–O bonding strength. It has been reported that the relative thermal stability of POE-metal complex was influenced by the polarization through different species of ionic M^{n+} .^{21,22} The relative thermal stability of metal-chelated polymers was examined using TGA (Fig. 3 and Table IV). The trend of thermal stability was observed in the order SMA-POE2000-LiCl > SMA-POE2000 (without metal ions) > SMA-POE2000-MgCl₂ ≈ SMA-POE2000-NiCl₂ > SMA-POE2000-CuCl₂ > SMA-POE2000-LiClO₄. In the overall trend, metal ions only slightly influenced polymer stability except for LiClO₄, which

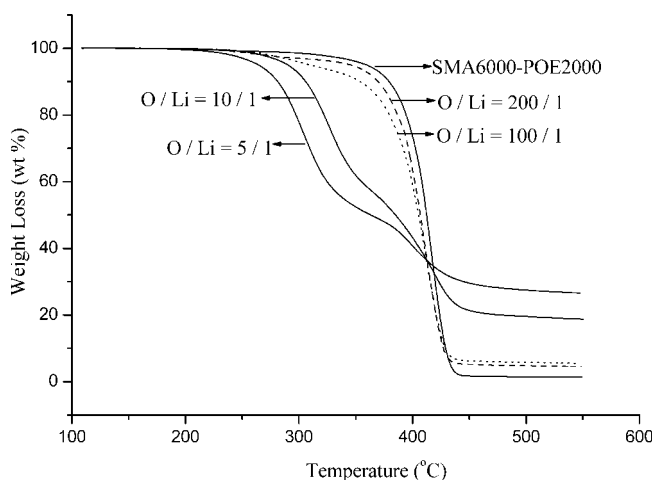


Figure 4 TGA of SMA-POE2000-LiClO₄ films containing varied amounts of Li⁺.

TABLE V
Comparison of TGA Decomposition Patterns for SMA-POE2000
Films with LiClO₄ and LiCl Doping

SMA-POE2000/ Li ⁺ O/Li ⁺	Temperature (°C) at				Char yield (%) at	
	95%	75%	50%	25%	450°C	500°C
	Retention (wt %)					
None	359	399	412	422	0	0
LiClO ₄						
200	345	392	407	417	5	5
100	311	386	405	420	6	4
10	290	327	386	433	22	20
5	265	303	363	— ^a	30	27
LiCl						
200	361	406	418	427	4	3
100	364	403	414	422	7	6
10	363	405	416	424	10	9
5	361	397	411	421	12	11

^a Char yield at 550°C = 26 wt %.

destabilized the polymer matrix with a TGA decomposition rate much faster than the pristine SMA-POE2000. It is important to understand the effect of metal ions on ionic conductivity and possible polymer destabilization in order to design a practical useful material. These relative decomposition trends are summarized in Table IV.

It is interesting that the variation in how the Li⁺ species influences polymer degradation. Figure 4 shows the TGA patterns of SMA-POE2000-LiClO₄ with (CH₂CH₂O)/Li⁺ molar ratios ranging from 200 to 5. It was determined that the starting SMA-POE2000 film in the absence of metal ions had higher stability than the polymer with added LiClO₄, by comparing their 95 wt % retention temperatures (359°C versus 265°C), as shown in Table V. With LiCl added at O/Li⁺ = 5 in SMA-POE2000-LiCl, polymer stability was not affected (361°C). Previously, we reported that POE decomposed through free-radical generation and a propagation mechanism.²³ The reason for the differences in the influences of the LiClO₄ and LiCl species in enhancing or inhibiting the fate of the radical species is not completely understood.^{18,24} It was observed the addition of an increasing amount of LiClO₄ substantially accelerated polymer decomposition. However, the low thermal stability for SMA-POE2000-LiClO₄ also indicated (CH₂CH₂O)/Li⁺ complexation through a separate ion pair was highly effective. For the ionic-conducting applications, which lithium ion species selected could eventually affect thermal stability.

CONCLUSIONS

A convenient method for preparing polymer films at low temperatures from SMA and POE diamine has been developed. Film materials consisting of POE

crosslinked networks had the advantages of low crystallinity and hence high electrostatic dissipating ability. Through the ability of POE to chelate metal ions, the polymers shifted their conducting mechanism from electrostatic to ionic conductivity, which was adjustable according to POE weight fraction and metal ion doping. For electrostatic dissipation, surface resistivity of 10^{6.8}Ω/sq was observed for the SMA-POE2000 film. With metal ion doping, the same film exhibited resistivity as low as 10^{4.1}Ω/sq. The process of instant mixing and film preparation produced a series of SMA-POE films that are potentially useful for both antistatic and ionic-conducting materials.

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