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Studies on blends of ionomers

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Blends of the zinc salt of poly(ethylene-*co*-methacrylic acid) (ZnEMA) and the zinc salt of sulfonated ethylene–propylene–diene monomer rubber (SEPDM), show thermoplastic elastomeric behaviour. The physical properties of the blends are greater than that predicted by the additivity rule. Results of dynamic mechanical analyses show ionic transitions in addition to T_g in the case of neat polymers and blends. Dielectric thermal analyses show that the activation energy due to ionic groups in dipolar relaxation is highest in the case of the 50/50 ZnEMA/SEPDM blend, presumably due to formation of interfacial ionic aggregates in the blend, evidence of which was obtained by infra-red spectroscopic studies. Reprocessability studies in the Monsanto processability tester show that the blend could be reprocessed (that is, extruded up to three cycles) like a thermoplastic without deterioration in strength. Copyright © 1996 Elsevier Science Ltd.

(Keywords: zinc salt of poly(ethylene-*co*-methacrylic acid); zinc salt of sulfonated ethylene–propylene–diene monomer rubber; blend)

INTRODUCTION

Ionic polymers wherein ionic domains act as physical cross-links exhibit attractive physical properties[1–5]. The morphology and properties of ionomers are dependent on the type of polymer backbone, ionic content, type of cation and the degree of neutralization[6–8]. The zinc salt of poly(ethylene-*co*-methacrylic acid) (ZnEMA) is a flexible thermoplastic ionomer which exhibits excellent tensile properties, abrasion resistance and clarity[3]. The zinc salt of sulfonated ethylene–propylene–diene monomer rubber (SEPDM) is an ionic elastomer[6]. It is known that sulfonate ionomers when compared under similar conditions possess much stronger ionic associations than the corresponding carboxylate ionomers[9]. The ionic associations are thermally reversible, permitting adequate melt flow at processing conditions[6,9].

There are several reports on blends of ionomers with other polymers. In blends of SEPDM with high-density poly(ethylene) (HDPE), SEPDM is reported to improve the impact and tensile strength of HDPE[10]. Blends of SEPDM with poly(propylene), poly(vinyl chloride), poly(ethylene oxide) and sulfonated butyl rubber exhibit unique characteristics[11–13]. Lu and Weiss have attributed the miscibility in blends of polyamide-6 and manganese sulfonated polystyrene to interpolymer interactions[14]. Interaction of acidic groups in one polymer (i.e. poly(styrene-*co*-styrene sulfonic acid)) with basic groups in another polymer (i.e. poly(ethyl acrylate-*co*-4-vinylpyridine)) to form ion pairs has been described as a route to compatibilization[15–18].

This paper reports the results of our studies on blends of the ZnEMA and SEPDM.

EXPERIMENTAL

Materials

The ZnEMA (obtained from E. I. Dupont de Nemours & Co., USA) had a carboxylate content of 10 meq (100 g polymer)$^{-1}$, a T_m of 95°C and a melt flow index of 5.5 g (10 min)$^{-1}$. The SEPDM (obtained from Exxon Research and Engineering Co., USA) contained 50 wt% ethylene and 5 wt% 5-ethylidene-2-norbornene. The sulfonation level was 25 meq (100 g polymer)$^{-1}$, and the Mooney viscosity, ML (1 + 4), at 100°C, was 20.

Blend preparation

The ZnEMA/SEPDM blends, 70/30, 50/50 and 30/70 (w/w), were prepared in a Brabender Plasticorder (model PLE-330) using cam-type rotors at a rotor speed of 60 r.p.m. and a temperature of 170°C. Prior to blending, ZnEMA was allowed to melt for about 2 min, and SEPDM was then added. The total mixing time was 7 min. The neat polymers were also masticated under the same conditions. The test specimens were prepared by moulding in an electrically heated hydraulic press for 5 min at 170°C. After moulding, the samples were immediately cooled down to room temperature by cold water circulation through the platens.

Blend characterization

Determination of physical properties. The stress–strain properties were determined at 25, 50 and 70°C according

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to ASTM D412 (1987) using dumb-bell test pieces in a Zwick Universal Testing Machine (UTM), model 1445, fitted with a temperature-controlled cabinet, using a cross-head speed of 500 mm min^{-1}. The tension set at 100% elongation was determined as per ASTM D412 (1987). The tear resistance was determined as per ASTM D624 (1986) using unnicked 90° angle test pieces (die C) at 25°C at a cross-head speed of 500 mm min^{-1} in a Zwick UTM (model 1445). The abrasion resistance was determined by a DuPont abrasion tester (BS 903: Part A9 1957, method C) and expressed as abrasion loss, which is the volume in cubic centimetres abraded from a test specimen per hour. Hardness was determined as per ASTM D2240 (1986) and expressed in Shore A units. The hysteresis loss was determined under strain mode according to ASTM D412 (1980) by stretching dumb-bell specimens to a strain level of 200% in a Zwick UTM (model 1445).

Dynamic mechanical analysis. Dynamic mechanical analyses of the samples (40 × 6.5 × 2 mm) were performed in a Rheovibron DDV-III-EP viscoelastometer at a frequency of 3.5 Hz and a strain amplitude of 0.0025 cm. The measurements were carried out over a temperature range of −100 to +200°C at a heating rate of 1°C min^{-1}.

Dielectric thermal analysis. Dielectric thermal analyses were done in a DuPont DEA 2970 Dielectric Analyser in a nitrogen environment. Square-shaped specimens of 25 mm side and 0.5 mm thickness were used. The measurements were carried out over a frequency range of 1000–100 000 Hz and a temperature range of 20–200°C at a heating rate of 5°C min^{-1}.

Infra-red spectroscopy. Infra-red spectroscopic studies of the compression-moulded thin films were carried out using a Perkin-Elmer 843 spectrophotometer with a resolution of 3.2 cm^{-1}.

Determination of reprocessability. The reprocessability of the 30/70 ZnEMA/SEPDM blend was studied by extruding the sample through a Monsanto processability tester (MPT) at 190°C, using a die of *L*/*D* ratio 20 at a shear rate of 18 s^{-1}. The extrudate was re-extruded under similar conditions, and the process was repeated for up to three cycles. Before each extrusion the sample was pre-heated in the barrel of the MPT at 190°C for 10 min. The apparent viscosity and tensile strength of the extrudate after each cycle were noted. The extrudate sample after each cycle was allowed to rest for 24 h before the tensile strength was measured.

RESULTS AND DISCUSSION

Physical properties

The physical properties of the neat polymers and the blends are summarized in *Table 1*. The stress–strain properties of the neat polymers and the blends at 25, 50 and 70°C are shown in *Figure 1*. The relationship between modulus at 300% elongation and composition of the blends at room temperature is shown in *Figure 2a*. The 70/30 and 50/50 ZnEMA/SEPDM blends show moduli which are very close to the additive values. The 30/70 ZnEMA/SEPDM blend shows a modulus which is higher than that calculated. The relationship between the room temperature tensile strength and composition of the blends is shown in *Figure 2b*. Synergism was observed in the sense that the observed tensile strengths of the blends were higher than the calculated averages. The 50/50 ZnEMA/SEPDM blend shows the highest tensile

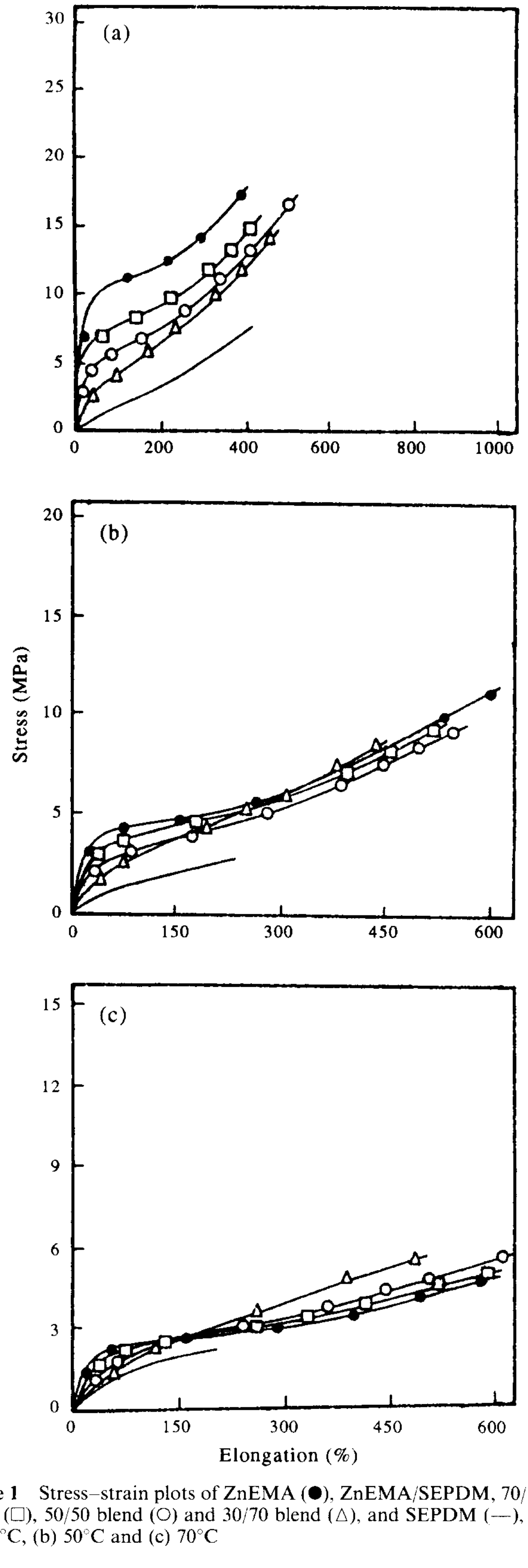

Figure 1 Stress–strain plots of ZnEMA (●), ZnEMA/SEPDM, 70/30 blend (□), 50/50 blend (○) and 30/70 blend (△), and SEPDM (—), at (a) 25°C, (b) 50°C and (c) 70°C

Table 1 Physical properties of the polymer samples at room temperature

Property	Blend composition (ZnEMA/SEPDM, w/w)				
	100/0	70/30	50/50	30/70	0/100
Modulus at 300% elongation (MPa)	14.0	11.3	9.6	9.2	5.1
Tensile strength (MPa)	17.4	15.3	16.2	14.9	7.5
Elongation at break (%)	408	438	509	483	425
Tear strength (kN m^{-1})	73	129	125	122	74
Hardness (Shore A)	80	83	81	73	54
Hysteresis loss (10^3 J m^{-2})	691	498	331	221	27
Tension set at 100% elongation (%)	46	24	13	8	2
Abrasion loss (Cm^3 h^{-1})	0.25	0.24	0.18	0.13	0.11

strength. The synergistic effect in the blends of the two ionomers may be attributed to the enhanced compatibility caused by the interpenetrating networks arising out of the interaction of the ionic groups present in the two components[12]. The blends showed higher elongation at break than that of either of the individual component polymers. The 50/50 ZnEMA/SEPDM blend showed the highest elongation at break. The blends showed better retention of stress–strain properties at higher test temperatures (*Figure 1*). At 70°C, for example, the blend compositions showed higher tensile strength and modulus as compared to the neat polymers.

Figure 2c shows the tear strength–composition relationship. The blends showed higher tear strength than the component polymers. Tear strength is a measure of the crack or slit propagation. It is known that tear strength is enhanced by factors which tend to dissipate energy[19]. The ionic domains present in both the ionomers and the crystallites present in ZnEMA could be considered as physical cross-links[20,21]. The ionic cross-links formed at the interfaces of the two polymers may be the reason for synergism in tear strength.

ZnEMA showed a higher hardness than SEPDM. *Figure 2d* shows the relationship between hardness and composition of the blends. The straight line represents the theoretical additive relationship. Blends show higher hardness than the calculated averages.

The results of hysteresis studies are shown in *Figure 3*. The hysteresis loss shown by the blend compositions were in between the hysteresis loss shown by the individual components. ZnEMA, because of its thermoplastic nature, shows very high tension set (*Table 1*) as compared to SEPDM. Tension set decreases as the

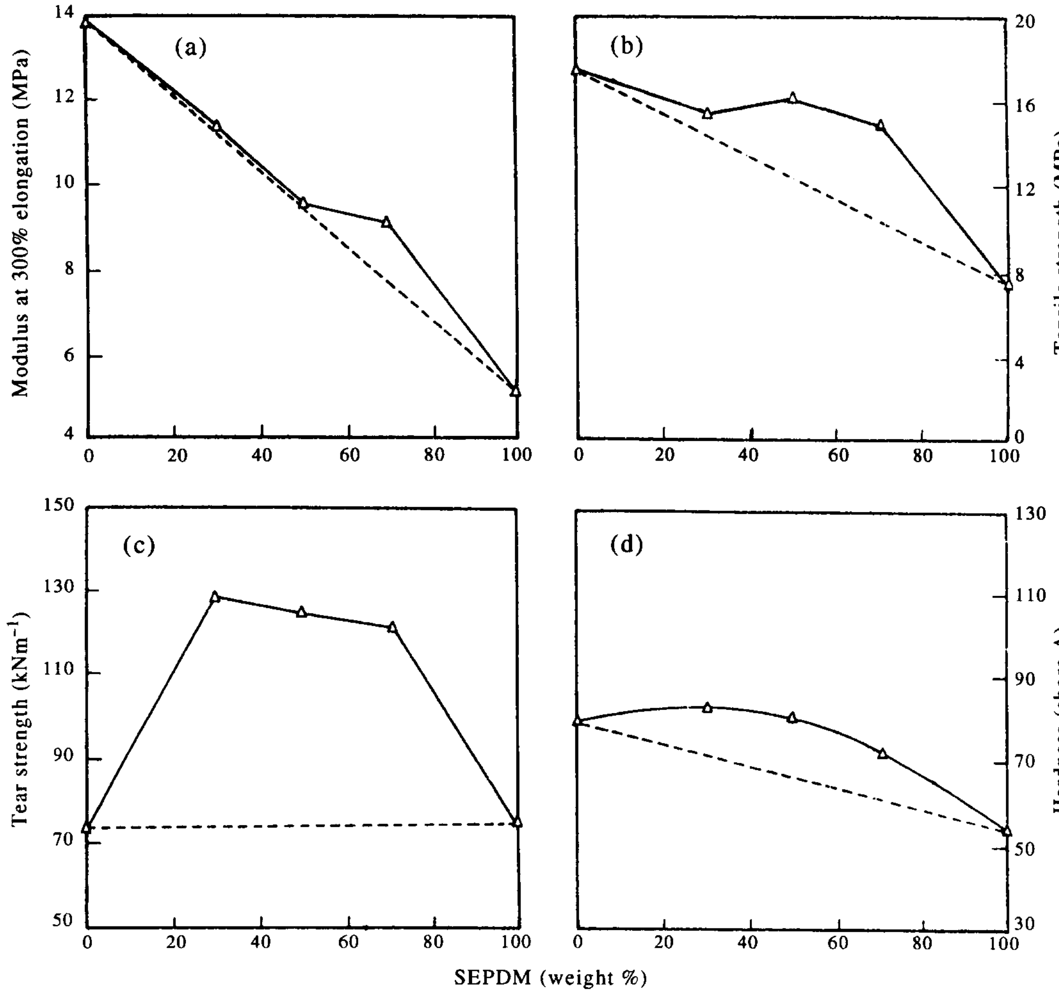

Figure 2 Variation of (a) modulus at 300% elongation, (b) tensile strength, (c) tear strength and (d) hardness with blend composition. △, observed values at room temperature; - - -, additivity line

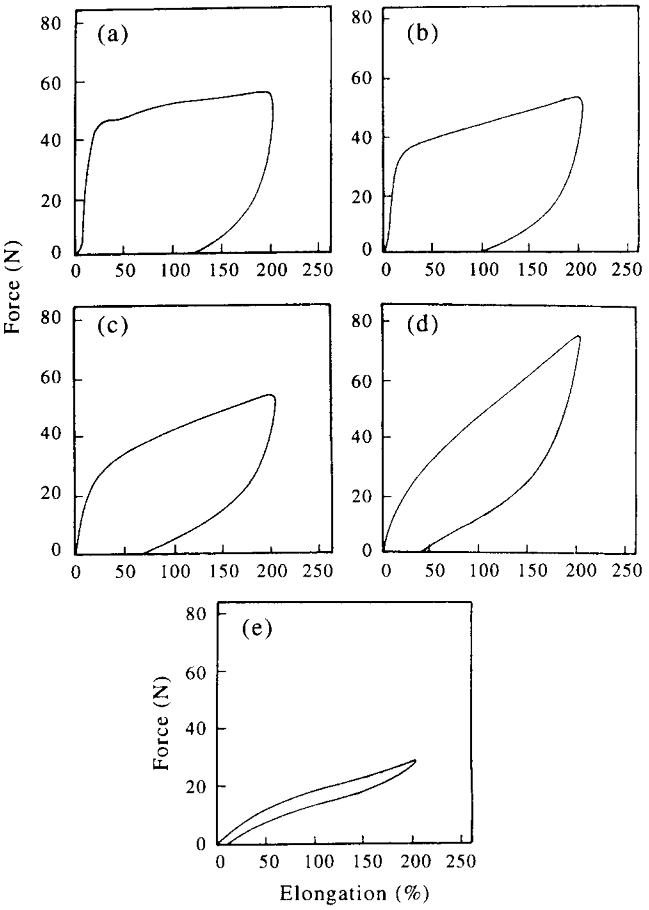

Figure 3 Hysteresis plots of (a) ZnEMA, ZnEMA/SEPDM, (b) 70/30 blend, (c) 50/50 blend and (d) 30/70 blend, and (e) SEPDM

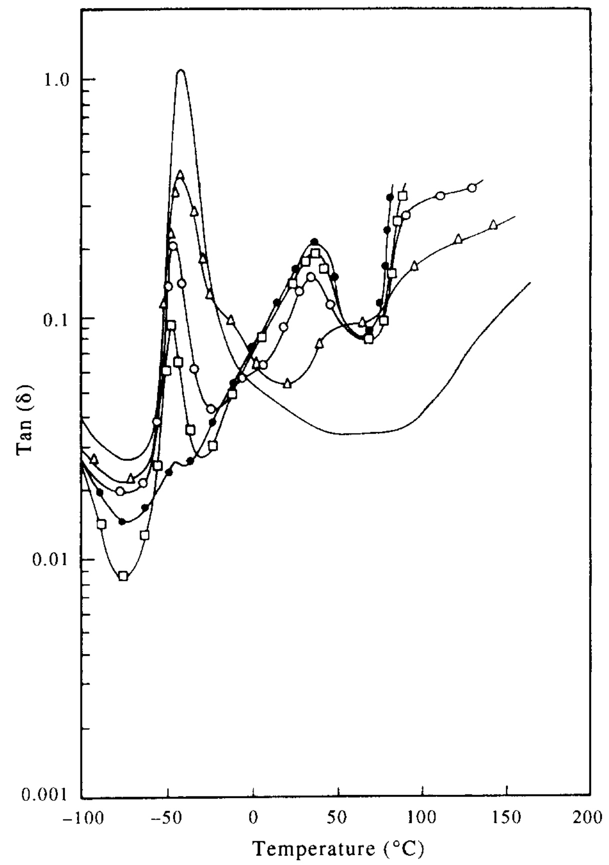

Figure 4 Semilogarithmic plots of tan δ *versus* temperature of ZnEMA (●) and ZnEMA/SEPDM, 70/30 blend (□), 50/50 blend (○) and 30/70 blend (△), and SEPDM (—)

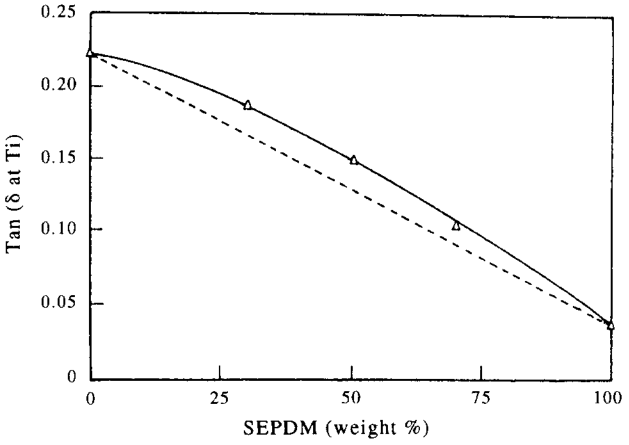

Figure 5 Variation of tan δ_{max} at T_i with blend composition. △, observed values; - - - , additivity line

percentage of SEPDM increases in the blend. SEPDM shows lower abrasion loss as compared to ZnEMA, as shown in *Table 1*. Abrasion loss decreases as the proportion of SEPDM increases in the blend.

Dynamic mechanical properties

Figure 4 shows the plot of loss tangent (tan δ) against temperature obtained from the dynamic mechanical analyses of neat polymers and the blends. ZnEMA shows two transitions, a weak transition at −59°C, which is due to the glass–rubber transition, and one at +38°C, which is presumably due to the occurrence of a rigid phase arising out of the ionic aggregates[22–24]. The low-temperature transition is denoted by T_g, and the high temperature transition by T_i. It has been reported earlier that in the case of partially ionized ZnEMA, the transition at T_g is suppressed by the transition at T_i[25]. The transition due to melting of the crystallites could not be detected due to softening of the sample. SEPDM shows T_g at −42°C and a broad transition (plateau) at T_i in the temperature range +38 to + 87°C. It has been reported earlier that in the case of crystalline ionomers, transition due to ionic aggregates occurs at a temperature lower than the crystalline melting temperature[26,27]. In the case of rubbery ionomers, the transition due to ionic groups has been found to occur in the same temperature range[28]. The T_g values of the component polymers were very close, and therefore the criterion of a single composition-dependent T_g could not be used to assess the miscibility of the blends[29]. Since the T_g of ZnEMA is not pronounced, T_g values of the blends appear close to that of SEPDM. The tan δ_{max} (that is, the tan δ value at T_g) was a maximum in the case of SEPDM and a minimum in the case of ZnEMA . In the blends, tan δ_{max} decreased as the concentration of ZnEMA increased, indicating stiffening[30]. The observed values of tan δ at T_i were higher than the calculated averages (*Figure 5*), indicating that the extent of ionic phase separation in the blend is greater than that predicted by

Table 2 Results of dynamic mechanical analyses

Blend composition (ZnEMA/SEPDM, w/w)	T_g (°C)[a]	tan δ at T_g (tan δ_{max})	T_i (°C)[b]	tan δ at T_i
100/0	−59	0.015	+37.6	0.219
70/30	−45	0.097	+36	0.193
50/50	−44	0.210	+35	0.151
30/70	−42	0.416	+52 to +73	0.097
0/100	−42	1.124	+38 to +87	0.038

[a] From $(\tan\delta)_{max}$ in the plot of tan δ *versus* temperature
[b] From tan δ *versus* temperature

the additivity rule. The results of dynamic mechanical analyses are summarized in *Table 2*.

Dielectric thermal properties

Figure 6 shows the tan δ *versus* temperature plots obtained from the dielectric thermal analyses of the neat polymers and the blends. ZnEMA showed transitions at 35, 51 and 78°C at 1000, 10 000 and 100 000 Hz, respectively. In the case of SEPDM the corresponding transitions occurred at 70, 81 and 92°C, respectively. Though the 70/30 ZnEMA/SEPDM blend showed the corresponding transitions at lower temperatures (that is, 34, 44 and 53°C), in the case of 50/50 and 30/70 ZnEMA/SEPDM blends the transitions occurred at higher temperatures (that is, 160, 163 and 168°C, and 142, 157 and 166°C, respectively). Thermal activation of dipolar relaxation generally follows the well-known Arrhenius law, where the activation energy for relaxation (E_{act}) could be calculated from the slope in the plot of log f *versus* $1/T_{max}$, where f is the frequency and T_{max} is the temperature corresponding to the maximum value of tan δ in the plot of tan δ *versus* temperature at any frequency[31,32]. *Figures 7* shows the Arrhenius plots of log(frequency) *versus* $1/T_{max}$. The activation energy of transitions due to ionic aggregates calculated from the slopes of Arrhenius plots are given in *Table 3*. SEPDM shows a higher activation energy than ZnEMA. This is because of the stronger ionic association in the sulfonate ionomer as compared to the carboxylate ionomer[9]. The activation energy followed the order ZnEMA < 70/30 ZnEMA/SEPDM = SEPDM < 30/70 ZnEMA/SEPDM < 50/50 ZnEMA/SEPDM.

These observations could be explained on the basis of the models for the distribution of salt groups in ionomers, which postulate the existence of multiplets and clusters[23,24,33,34]. Multiplets are aggregates consisting of ion pairs distributed in the matrix. When sufficient numbers of multiplets are close enough together to form a contiguous region of restricted mobility greater than 50–100 Å in size, the region constitutes a cluster and exhibits its own separate T_g. The low activation energy observed in the case of the neat polymers and the 70/30 ZnEMA/SEPDM blend might be attributed to the smaller aggregates in clusters present in the matrix, and the high activation energy observed in the case of the 50/50 and 30/70 blends might originate from the larger aggregates in the clusters[22]. Formation of aggregates at the interphases of the two polymeric phases may lead to formation of larger aggregates, particularly in the 50/50 ZnEMA/SEPDM blend.

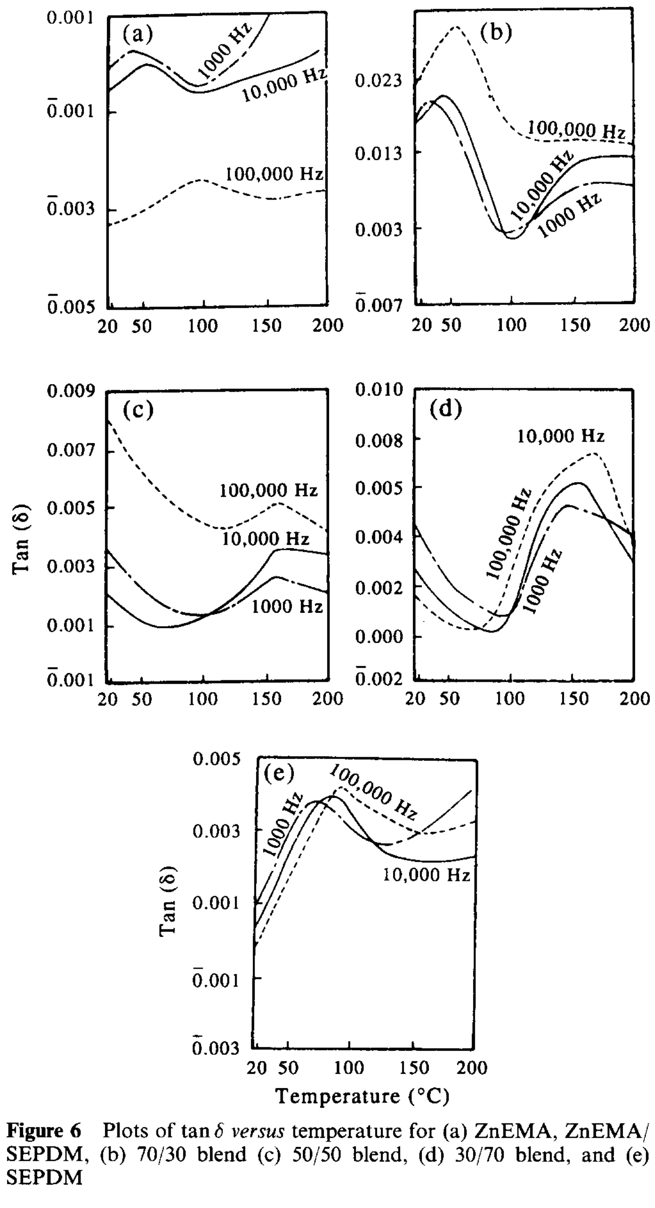

Figure 6 Plots of tan δ *versus* temperature for (a) ZnEMA, ZnEMA/SEPDM, (b) 70/30 blend (c) 50/50 blend, (d) 30/70 blend, and (e) SEPDM

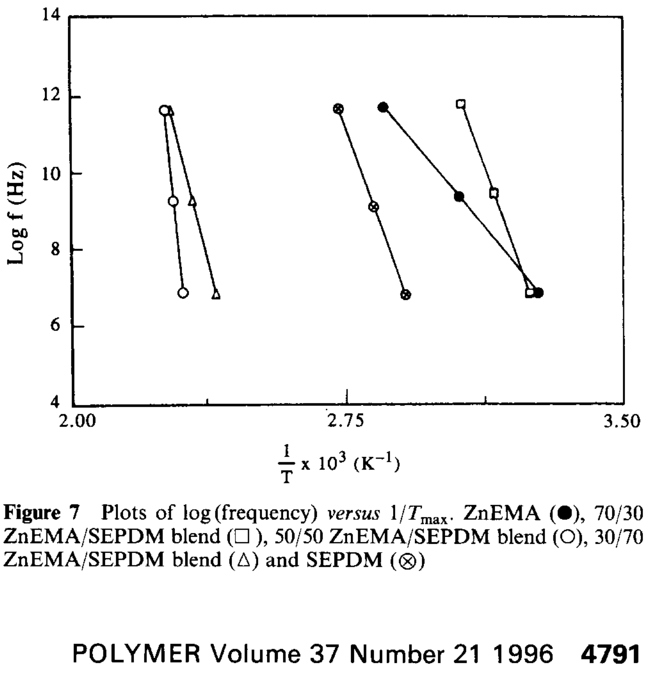

Figure 7 Plots of log(frequency) *versus* $1/T_{max}$. ZnEMA (●), 70/30 ZnEMA/SEPDM blend (□), 50/50 ZnEMA/SEPDM blend (○), 30/70 ZnEMA/SEPDM blend (△) and SEPDM (⊗)

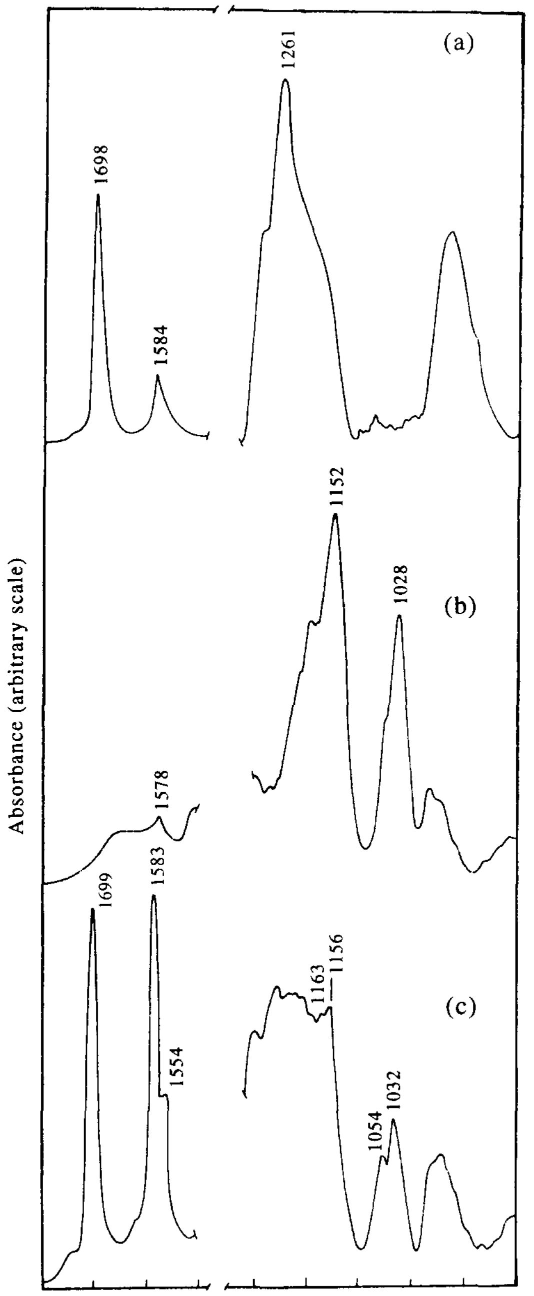

Figure 8 Infra-red spectra of (a) ZnEMA, (b) SEPDM and the (c) 50/50 ZnEMA/SEPDM blend

Infra-red spectroscopic analysis

Infra-red spectra of ZnEMA, SEPDM and the 50/50 blend are shown in *Figure 8*. The spectra of ZnEMA shows a band for C=O stretching, corresponding to hydrogen-bonded carboxyl groups, around 1698 cm^{-1}. The carboxylate asymmetric stretching band for bridging metal carboxylate occurs around 1584 cm^{-1}. The strong broad band in the 1300–1100 cm^{-1} region with a peak at 1261 cm^{-1} is believed to be due to a C–O stretching vibration[35–37].

In SEPDM the S=O asymmetric stretching vibration band appears at 1152 cm^{-1}. However, this peak is assigned to the composite absorption effect of S=O asymmetric stretching vibration of sulfonate anions. The S = O symmetric stretching vibration of sulfonate ions shows a distinct sharp band at 1028 cm^{-1}. In the SEPDM, the peak at 1578 cm^{-1} is due to stretching of the C=C system with a sulfonyl group[35,36,38].

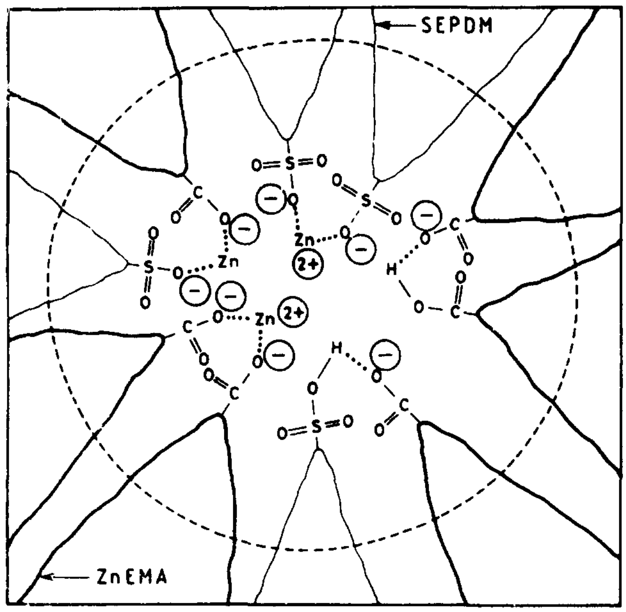

Figure 9 Schematic representation of the interaction between ZnEMA and SEPDM

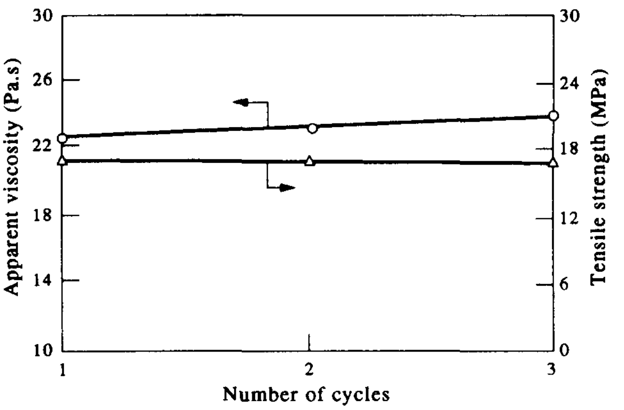

Figure 10 Apparent viscosity at 190°C and tensile strength of the extrudate at 25°C of the 30/70 ZnEMA/SEPDM blend at different cycles of extrusion through the MPT

It is seen from the spectra of the 50/50 ZnEMA/SEPDM blend that the C=O stretching peak of hydrogen-bonded COOH groups is not changed in position, indicating that the effect of hydrogen bonding to be of little importance.

The carboxylate asymmetric stretching band occurs at a lower frequency (in place of 1583 cm^{-1}), and shows a

Table 3 Activation energies for ionic transitions from dielectric thermal analyses

Blend composition (ZnEMA/SEPDM, w/w)	Activation energy (kcal mol^{-1})
100/0	22.9
70/30	51.2
50/50	182.0
30/70	75.0
0/100	54.2

[a] Calculated from the plots of log (frequency) *versus* $1/T_{max}$

double peak at 1583 and 1554 cm^{-1}. The high intensity of the peak at 1583 cm^{-1} is possibly due to the composite absorbance of COO^- asymmetric stretching and $C = C$ stretching[35–38]. The appearance of a double peak indicates changes in the geometrical arrangement of COO^- groups around a central Zn^{2+} ion, thereby suggesting participation of sulfonic acid groups as well as sulfonate salts in ionic aggregate formation along with the carboxylates.

In the blend system the $S = O$ asymmetric stretching band appears as a doublet at 1156 and 1163 cm^{-1} in a broad background. The broad background could possibly be due to the strong composite absorption of the $S = O$ and $C - O$ systems. On the other hand, the $S = O$ symmetric stretching vibration band which appears at 1028 cm^{-1} shows the existence of a doublet at 1032 and 1054 cm^{-1}. The band width at half height of the $S = O$ symmetric stretching band is also broadened in the blend. The larger bandwidth clearly suggests that the $S = O$ absorption frequency threshold has been shifted to the higher-frequency region, thus indicating the possibility of formation of a stronger ionic field in the blend[39]. The whole change in the spectral feature is believed to be due to the effect of some new ionic groups which participate in cluster formation together with the sulfonate ions.

It is inferred that in the blend the ionic groups of the polymers interact with one another, presumably at the interphases. A schematic representation of the formation of ionic aggregates is shown in *Figure 9*. Synergism observed in the physical properties is believed to be due to technological compatibility arising out of the formation of interfacial aggregates.

Reprocessability

Figure 10 shows the variation of apparent viscosity and tensile strength of the extrudate of the 30/70 ZnEMA/SEPDM blend with the number of cycles in extrusion through the MPT. It was observed that the apparent viscosity of the blend and the tensile strength of the extrudate remain almost constant by repeated preheatings and extrusions. This shows that the ZnEMA/SEPDM blend behaves as a thermoplastic elastomer and could be reprocessed by mechanical recycling without deterioration of its properties.

CONCLUSIONS

Blends of ZnEMA and SEPDM behave as thermoplastic elastomeric ionomers and show modulus, tensile strength, tear strength, hardness and abrasion resistance properties which are higher than the calculated averages. Tension set decreases with increasing SEPDM content in the blends. Dynamic mechanical analyses show an ionic transition at T_i in addition to a glass–rubber transition at T_g in the case of neat polymers and blends. The observed $\tan\delta$ at T_i for the blends was higher than the calculated averages. Dielectric thermal analyses show that the activation energy for dipolar relaxation in the ionic transition region is highest in the case of the 50/50 ZnEMA/SEPDM blend, presumably due to maximum ionic cluster formation at the interphases. Infra-red spectroscopic studies provide evidence for the interaction of ionic groups present in the blend components, giving credence to the results of dynamic mechanical and dielectric thermal analyses. Reprocessability studies in the MPT show that the ZnEMA/SEPDM blend behaves as a thermoplastic ionomer and could be reprocessed by mechanical recycling without deterioration in its properties.

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