

Mechanical and Thermal Properties of Poly(vinyl chloride)/ α -Methyl-styrene-acrylonitrile Blends Prepared by Melt Extrusion

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ABSTRACT: In this article, we have examined the physical and mechanical properties of poly(vinyl chloride) (PVC)/ α -methyl-styrene-acrylonitrile (α MSAN; 31 wt % AN concentrations) blends with different blend ratios. And, we also examined the effect of the molecular weights of PVC on the miscibility and material properties of the blends prepared by melt extrusion blending. Our results showed that the PVC/ α MSAN blends have good processing properties and good miscibility over all blend ratios because of the strong interac-

tion between PVC and α MSAN. And, the blends showed enhanced mechanical and thermal properties. In addition, high molecular weight PVC showed reasonable processability when melt blended with α MSAN, which resulted in enhanced mechanical and physical properties. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 111: 237–245, 2009

Key words: PVC; α MSAN; high molecular weight PVC; blends; mechanical properties; melt extrusion

INTRODUCTION

Poly(vinyl chloride) (PVC) is known as a versatile thermoplastic with good mechanical properties and also possesses excellent flame retardant properties among general purpose commercial plastics.^{1–4} However, its thermal stability is not always satisfactory because of its low glass transition temperature (T_g). A formulation that performs well at room temperature application may become miserable even for 80°C environments. For amorphous polymers or semicrystalline polymers with low crystallinity (PVC usually has 5–20% crystallinity), thermal properties such as the heat distortion temperature (HDT) and the Vicat softening temperature (VST) primarily depend upon its T_g .^{5–9} However, addition of reinforcing fillers (e.g., glass fiber) does not show significant improvement on the T_g in case of amorphous polymers or semicrystalline polymers with low crystallinity.⁸ Therefore, the plausible way of improving T_g of these polymers would be to blend PVC with a high T_g polymer.

To be used in blending, the polymer should be compatible with PVC to reduce the cause of any processing complications shown in incompatible blends. In this case, it is extremely important to control the domain size to have expected physical properties.¹⁰ Additionally, the rheological properties

should remain within an acceptable range for better dispersion.

A lot of studies on PVC blending have been accomplished by other groups.^{11–16} Among them, we have focused on the α MSAN/PVC blends. α MSAN in a wide range of acrylonitrile (AN) contents are miscible with PVC through solution blending. The intermolecular interaction between α MSAN and PVC is influenced by the AN contents in α MSAN. The intramolecular repulsive interaction between alpha methyl styrene and AN units of α MSAN and the additional intermolecular interaction between α -hydrogen of PVC and AN of α MSAN have accounted for the miscibility improvement of this blend system.¹⁷ In this blending system, two interactions exist; one is induced dipole interaction between aromatic quadruple (benzene ring) of α MSAN and halogen (chlorine), and the other is hydrogen bonding between AN and α -hydrogen of PVC. Methyl groups in α MSAN inhibit the main chain-rotation to help AN exposure. Compared to SAN, α MSAN shows improved miscibility with PVC in wide AN contents range due to gauche conformation of chain, which helps hydrogen bonding between AN and α -hydrogen of PVC. The interaction between PVC and α MSAN is detailed in our previous study.¹⁸ In the previous research, we could know that α MSAN with AN contents at 31 wt % exhibits a compatible morphology with PVC even through the melt extrusion blending.

First report on PVC/ α MSAN blend can be found in late 1980s.^{19–21} Modification of SAN was pursued

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for better miscibility with PVC. It has been specifically designed to increase the HDT (T_g) of general purposes thermoplastics including PVC without major adverse effect on other physical properties. Kim et al.¹⁷ found that blends of α MSAN having 30 wt % AN contents is miscible with PVC to show a single T_g for all composition by melt blending. Furthermore, no phase separation on heating did occur prior to 250°C, which is beyond the normal processing range for PVC. Another Kim et al.²² have studied a broad range of AN contents in α MSAN to show that the miscibility window is ranged from 16–51 wt % AN contents. Otherwise it is immiscible with PVC. Gan et al.¹³ have also found that blends of α MSAN with PVC showed a single T_g containing an AN level of 11.9 to 30 wt % with a sample obtained from a solution precipitation.

In this article, we have investigated the mechanical and physical properties of miscible PVC/ α MSAN with various blend ratios prepared by melt extrusion. We also investigated effect of molecular weight on the rheological, mechanical, and physical properties of PVC/ α MSAN blends PVC for further improvement in thermal property.

EXPERIMENTAL

Materials

Commercially available PVC and α MSAN (31 wt % AN) were obtained from LG Chemical Co. (Seoul, Republic of Korea) and BASF (Ludwigshafen, Germany), respectively, and were used as received. The physicochemical properties of these polymers are listed in Table I. To prepare basic PVC powder compound (P1), PVC resins and additives were mixed thoroughly in a turbo mixer at 800 rpm until it reached 120°C, and they were cooled to 60°C. To prevent degradation of the PVC, 2 wt % of tin maleate was added as a heat stabilizer in P1. Internal lubricants (0.5 wt % of calcium stearate and 0.5 wt % of fatty alcohol) and external lubricants (0.5 wt % of stearic acid and 0.5 wt % of oxidized polyethylene

TABLE I
Characteristics of the Polymers

Resin	AN (%)	M_w^b	T_g (°C)	MI (g/10 min)	Source
PVC 1000 ^a	–	140,000	74.5	4.4 ^c	LG CHEM.
PVC 1300 ^a	–	163,000	76.0	2.0 ^c	LG CHEM.
PVC 1700 ^a	–	205,000	76.4	0.5 ^c	LG CHEM.
α MSAN	31	95,000	125	14 ^d	BASF.

^a T_g and MI of PVC were evaluated from the rigid PVC composition (P1).

^b Weight average molecular weight.

^c Measured at 210°C under a 12.5-kg load.

^d Measured at 220°C under a 10-kg load.

TABLE II
Blend Ratios of P1, α MSAN, and P1/ α MSAN Blends with Different Blend Ratios

Sample	Blend ratio
P1	100 wt % PVC DP 1000 and additives
P1aM10	90 wt % P1/10 wt % α MSAN
–	–
P1aM90	10 wt % P1/90 wt % α MSAN
aM	100 wt % α MSAN

wax) were also added to improve the gelation of the PVC matrix and reduce the friction between powder particles and the extruder wall. All these processing considerations are the same as before.¹⁸

P1/ α MSAN blends with various blend ratios and different PVC molecular weights

According to the blend ratios, α MSAN pellets were mixed with the preprepared P1. Sample codes with different blend ratios are listed in Table II. To investigate the effect of molecular weight of PVC, one blend composition as 30 wt % α MSAN is chosen, and sample codes with different molecular weight of PVP are given in Table III.

Melt-extrusion blending and specimen preparation

The blend compounds were extruded using a Buss (Prattein, Switzerland) MDK46 kneader extruder (length/diameter 5/11) with a zone temperature profile of 170 ~ 190°C at 700 rpm. The extrudate was quenched in water and pelletized with a cutter, and dried in an oven. Specimens for mechanical and physical properties tests were molded by PVC injection molding machine with a zone temperature profile of 170 ~ 200°C in the LG Chemical Tech. Center in Korea.

Characterizations

A Haake rheometer (model 600, Karlsruhe, Germany) was used to study the melt rheology of the blends for the prediction of the processing properties.^{23–29} The melting torque and equilibrium melting torque were measured using this device. They were

TABLE III
Blend Ratio of P1, P1/ α MSAN Blends with Different Molecular Weight of PVC

Sample	Blend ratio
P1	100 wt % PVC DP 1000 and additives
P100aM30	70 wt % PVC DP 1000 and additives/30 wt % α MSAN
P130aM30	70 wt % PVC DP 1300 and additives/30 wt % α MSAN
P170aM30	70 wt % PVC DP 1700 and additives/30 wt % α MSAN

determined at 180°C and 30 rpm. The degradation time was also measured at 210°C and 30 rpm. A melt indexer (model F-B01, Toyoseiki, Tokyo, Japan) was used to measure the melt index (MI). It was determined at 210°C under a 12.5-kg load after 5 min of preheating.

A tensile tester (model UTM 4204, Instron, MA) was used to study the tensile strength of the blends according to ASTM D638. It was determined at an extension rate of 50.0 mm/min at 25°C with 50% relative humidity. The flexural strength/modulus testing was performed using an electronic tensile tester (model UTM 1425, Zwick, Ulm, Germany) according to ASTM D790. It was also determined at an extension rate of 50 mm/min at 25°C with 50% relative humidity.

A specific gravity (density) testing was performed according to ASTM D792. The Vicat softening temperature (VST; model S-6M2, Toyoseiki, Tokyo, Japan) was measured according to ASTM D1525 under a load of 1 kg. The temperature was elevated at a rate of 120°C per hour. T_g was determined using a differential scanning calorimetry (DSC; model 910, DuPont, Wilmington, USA). A heating rate of 10°C/min was used. Each sample was initially heated to 200°C and then rapidly quenched until the temperature was -20°C. The transition behavior was recorded during a second heating with the mid point of the transition defined as the T_g of the blend. UL 94 method was used for evaluating the flame retardancy of the blends. In the procedure, a specimen is supported in a vertical position, and a flame is applied to the bottom of the specimen. The flame is applied for 10 s and removed until flaming stops. Then, the flame is reapplied for another 10 s and removed.

The morphology was observed by scanning electron microscopy (SEM; model JSM 5610, Jeol Co., Tokyo, Japan). Samples were prepared using two methods: cryogenic fracturing of specimens prepared by a

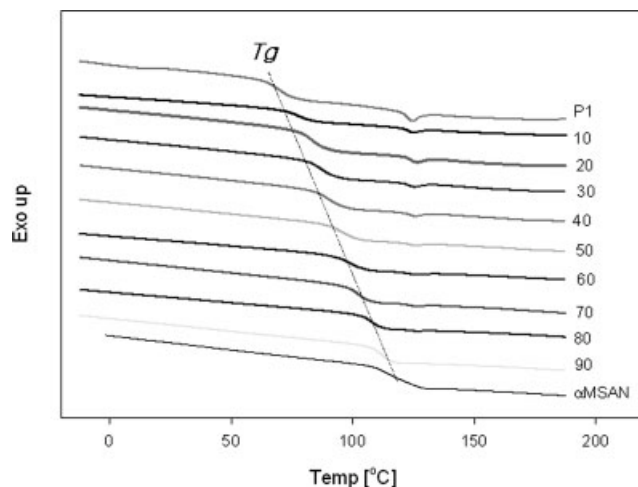


Figure 1 DSC thermodiagrams of P1, α MSAN, and P1/ α MSAN blends with different blend ratios.

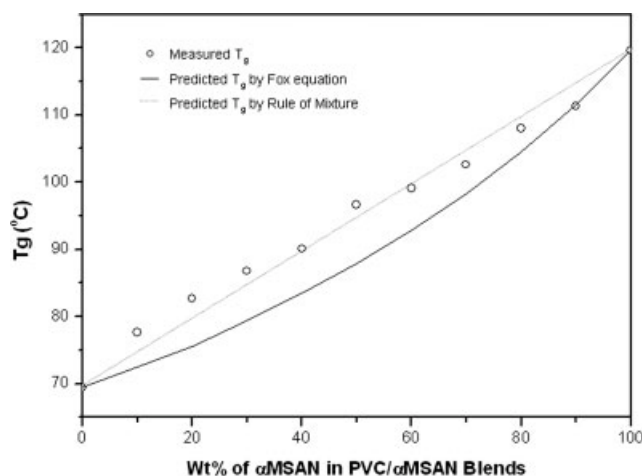


Figure 2 T_g values measured with DSC and predicted by the Fox equation of P1/ α MSAN blends with different blend ratios.

compression molding method and impact fracturing of specimen prepared by injection molding. To reconfirm the miscibility in the melt state, the rheological properties of the blends were measured with an advanced rheometrics expansion system (ARES; Rheometric Scientific, Leatherhead, UK). Press-molded disk specimen in the plate-and-plate fixture was used for the rheological measurement. The frequency sweep was done at 200°C and 10% strain, which is the upper limit for the linear visco-elastic behavior.

RESULTS AND DISCUSSION

Miscibility of P1/ α MSAN blends with different blend ratios

The miscibility of P1/ α MSAN blends with different blend ratios was investigated with DSC. The selective DSC thermo diagrams shown in Figure 1 indicate that the blends of P1/ α MSAN have a single T_g in over all blend ratios, which is consistent with the previous report.¹⁷ Figure 2 shows the measured of T_g values of P1/ α MSAN blends with different blend ratios. These values are slightly deviated from predictions made by the Fox equation.* Instead, the experimental data seem to adjust to a simple Rule of

*Fox equation:

$$\frac{1}{T_{g\text{mix}}} = \frac{x_a}{T_{g_a}} + \frac{x_b}{T_{g_b}}$$

Rules of Mixtures:

$$T_{g\text{mix}} = T_{g_a} \times x_a + T_{g_b} \times x_b$$

Here, $T_{g\text{mix}}$, T_{g_a} , and T_{g_b} are the T_g of the blend, polymers a and b, respectively. The weight fractions of each polymer are represented by x_a and x_b .

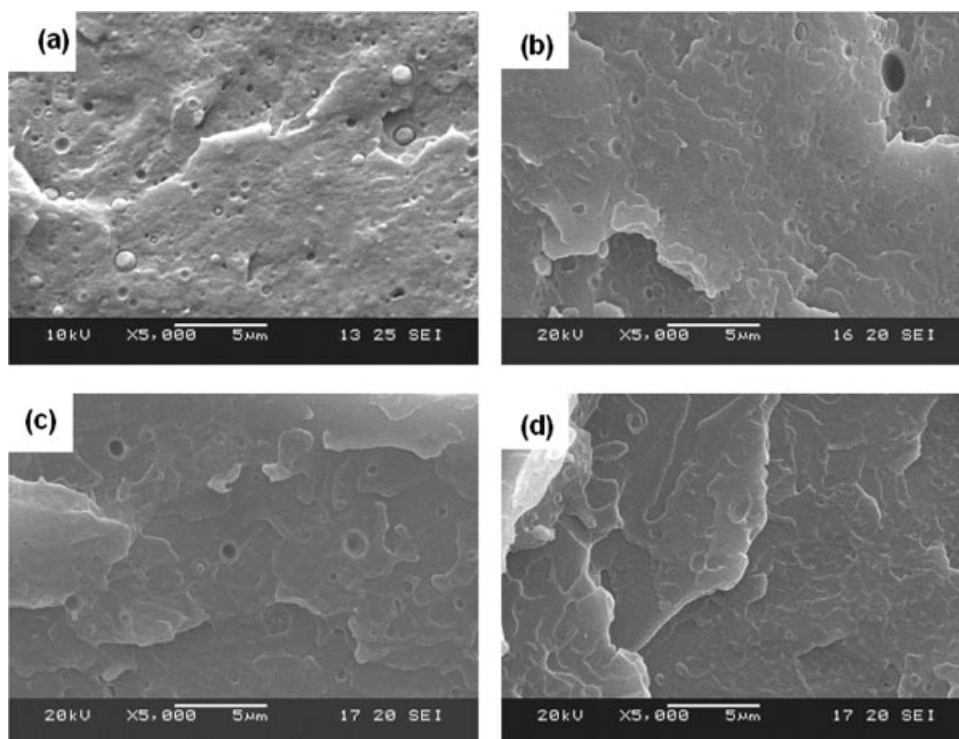


Figure 3 SEM images of cryogenically fractured surface of PVC and blends sample prepared by compression molding; (a) PVC (P1), (b) P1/ α MSAN 10 wt %, (c) P1/ α MSAN 30 wt %, and (d) P1/ α MSAN 50 wt % (Fig. 3(a) is a previously published figure.¹⁸).

Mixture,* which indicates that PVC/ α MSAN blends are completely miscible over all blend ratios.

The thermal analyses indicate that α MSAN has interactions strong enough to be mixed completely with PVC, even through by melt extrusion. α MSAN has a steric effect on α -methyl groups in the styrene units. The steric effect causes the copolymer chain to become more rigid, and inhibits the self-association of polar AN segments within the copolymer, and enables additional interactions between the α -hydrogen of PVC and the AN of α MSAN.

As shown by the cryogenically fractured surface of the blends sample by compression molding (Fig. 3), and as shown by the impact-fractured surface of samples by injection molding (Fig. 4), P1 and the P1/ α MSAN blends are brittle material and exhibited brittle breakage. P1 has a clear-cut surface and a nongelling part shown as a small and round particle in the SEM images. As α MSAN contents are increased, the fracture surface of the P1/ α MSAN blend becomes clearer than those of P1. This indicates that α MSAN has such good miscibility with PVC that it subsequently makes the PVC/ α MSAN blends' matrix more homogeneous than those of the PVC itself. As shown by the toluene-etched surface of the blends sample by compression molding (Fig. 5), the morphology of the P1/ α MSAN blends with different blend ratios shows similar characteristics.

The samples were put in toluene for 1 min. P1/ α MSAN blends show homogeneous morphology of up to 50% contents of α MSAN. The matrix is PVC/ α MSAN blends, and domains which consist of unmixed α MSAN particles and nongelling PVC particles are distributed in a circular shape with a small size of under 0.5 μ m. As the contents of α MSAN increase, the small domain increases, but phase separation does not occur in sea and island morphology.

The rheological properties of P1, α MSAN, and the P1/ α MSAN blends were measured with ARES. The storage modulus (G') and loss modulus (G'') are shown as functions of the frequency in Figure 6. G' is depending upon several factors such as the polarity, molecular weight (M_w), molecular weight distribution (MWD), chain stiffness, and dispersion morphology and so on. Because the M_w and MWD of the polymer used in this study do not differ greatly, the polarity, chain stiffness, and morphology could be responsible for different rheological properties. As shown in Figure 6(a), P1 has high G' of P1 may be because of the high polarity and heterogeneous morphology in the PVC itself. On the other hand, α MSAN has a low G' because of low polarity and molecular weight. P1/ α MSAN blends only show an average value between PVC and α MSAN. Loss modulus also exhibits a similar trend.

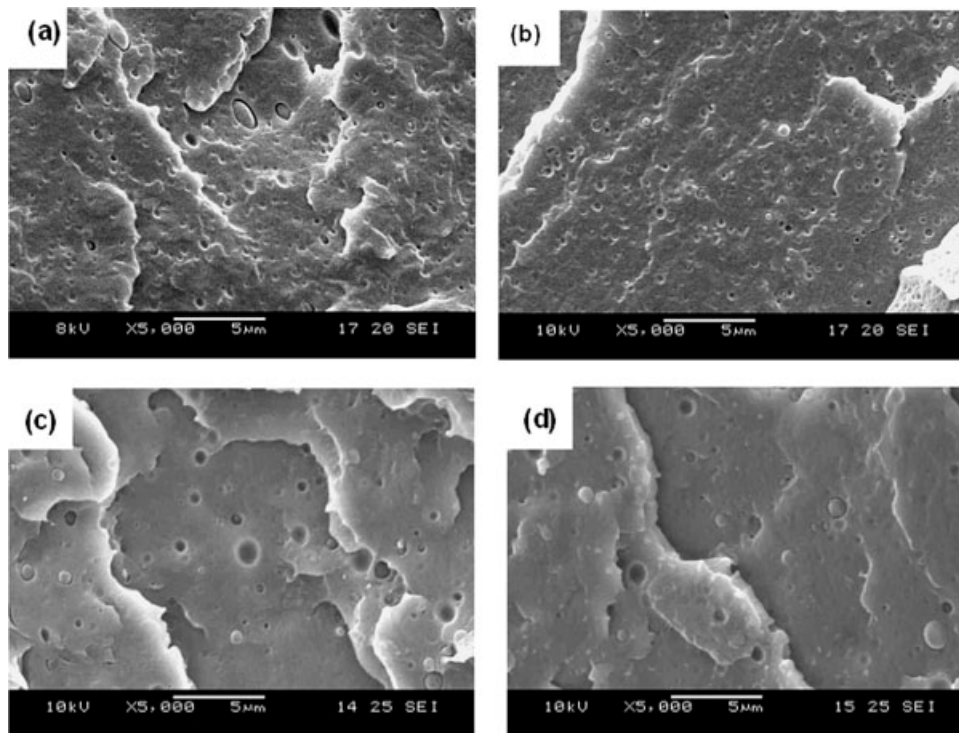


Figure 4 SEM images of the impact-fractured surface of PVC and blends sample by injection molding; (a) PVC (P1), (b) P1/ α MSAN 10 wt %, (c) P1/ α MSAN 30 wt %, and (d) P1/ α MSAN 50 wt % (Fig. 4(a) is a previously published figure¹⁸).

The results shown in the Figure 6(c) indicate that the P1/ α MSAN blends show more homogeneous behavior compared to the PVC in a molten state. It

is well known that if the polymers are homogenous, the slope of G'/G'' is close to 2. However, the slope is lower than 2 when the blend is immiscible or

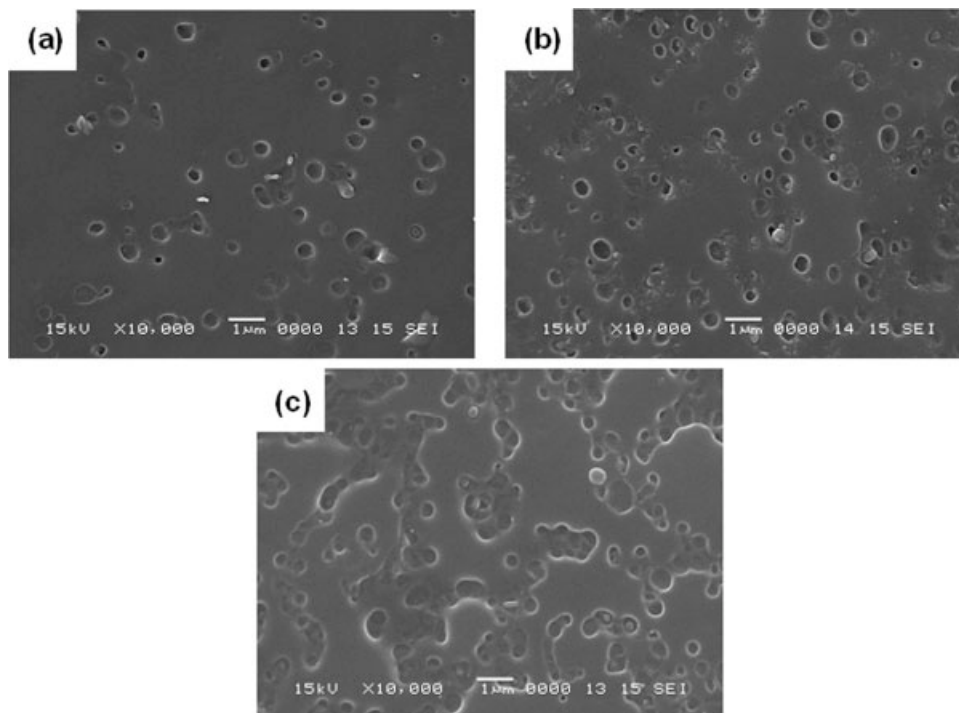


Figure 5 SEM images of toluene-etched surface of blends sample by compression molding; (a) P1/ α MSAN 10 wt %, (b) P1/ α MSAN 30 wt %, and (c) P1/ α MSAN 50 wt %.

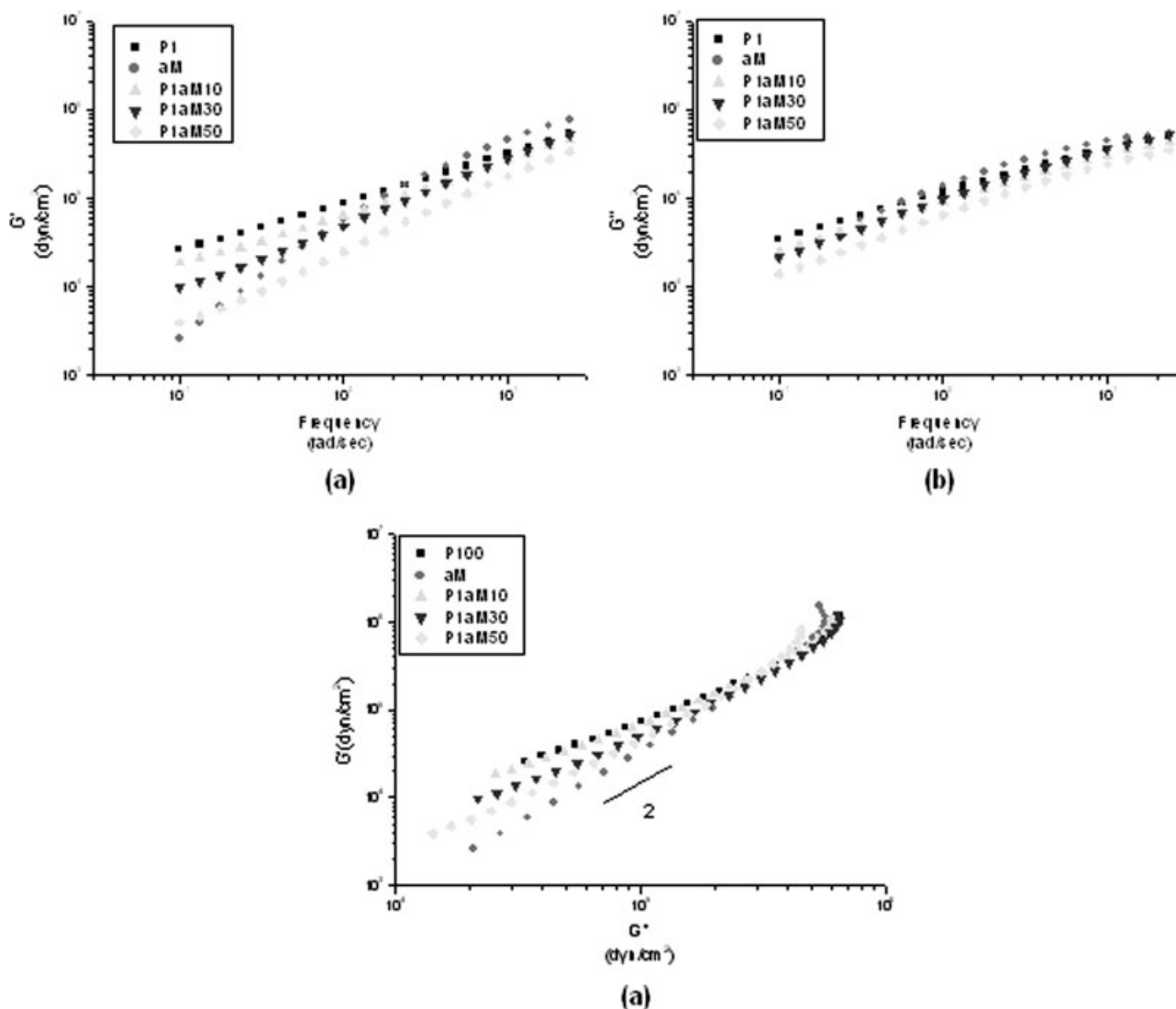


Figure 6 G' and G'' of the polymers and blends; (a) G' of PVC (P1), α MSAN (aM), P1aM10, P1aM30 and P1aM50, (b) G'' of PVC (P1), α MSAN (aM), P1aM10, P1aM30 and P1aM50, and (c) G' and G'' of PVC (P1), α MSAN (aM), P1aM10, P1aM30 and P1aM50.

heterogeneous.³⁰ As α MSAN contents increase, the slope of P1/ α MSAN blends becomes close to 2, which indicates that the blends have a more homogeneous morphology than that of PVC. The slope of the P1 shows that PVC is not homogeneous compared to the α MSAN copolymer. This may be caused by the existence of PVC nongelling particles in the PVC matrix.

Mechanical and thermal properties of P1/ α MSAN blends with different blend ratios

For amorphous polymers or semicrystalline polymers with low crystallinity, mechanical and thermal properties depend upon its T_g . In Figure 2, the experimentally measured T_g values of P1/ α MSAN blends increased linearly as the content of α MSAN increases. Therefore, it is expected that the mechani-

cal and thermal properties of the blends would be significantly improved according to the content of added α MSAN. The mechanical properties of P1/

TABLE IV
Mechanical Properties of P1, α MSAN, and P1/ α MSAN Blends with Different Blend Ratios

Sample	Tensile strength (MPa)	Flexural strength (MPa)	Flexural modulus (MPa)
P1	85.3	112.7	2842
P1aM10	87.7	115.6	2871
P1aM20	92.1	119.6	2871
P1aM30	93.6	122.5	2920
P1aM40	95.1	126.4	2842
P1aM50	88.2	127.4	2803
aM	102.9	142.1	2881

For each test, two sets of five specimens were tested and the average value was obtained.

TABLE V
Physical Properties of P1, α MSAN, and P1/ α MSAN Blends with Different Blend Ratios

Sample	Density (g/cm ³)	MI (g/10 min)	Vicat (°C)	UL94
P1	1.36	9.8 ^a	81.5	V-0
P1aM10	1.34	12.3 ^a	83.3	V-0
P1aM20	1.32	13.1 ^a	84.5	V-0
P1aM30	1.30	14.0 ^a	86.8	V-0
P1aM40	1.28	14.7 ^a	90.3	V-0
P1aM50	1.26	14.5 ^a	93.9	V-0
aM	1.18	14.0 ^b	110	V-2

^a Measured at 210°C under a 12.5-kg load.

^b Measured at 220°C under a 10-kg load.

For each test, two sets of five specimens were tested and the average value was obtained.

α MSAN blends with different blend ratios are given in Table IV. Blend ratios do not have a significant effect on the flexural modulus because these two polymers have almost same modulus originally; however, both tensile and flexural strength are linearly improved with the increasing α MSAN content. These results also indicate that the good miscibility of P1/ α MSAN blends has sufficiently beneficial effect on the mechanical properties.

The other physical properties of the P1/ α MSAN blends with different blend ratios are given in Table V. As expected, the VST increases linearly with the

TABLE VI
 T_g of P1/ α MSAN Blends with Different Molecular Weights of PVC

Sample	T_g by DSC (°C)
P100aM30	86.8
P130aM30	87.6
P170aM30	87.1

For each test, two sets of five specimens were tested and the average value was obtained.

increasing α MSAN content just as T_g s of the blends. On the contrary, the density decreases linearly according the content of α MSAN. The MI increases up to about 40% as α MSAN content increases and is maintained at 14 g/10 min of the level of α MSAN. In addition, the blends keep good flame retardant properties (UL 94 V-0) up to 50 wt % contents of α MSAN.

Miscibility of P1/ α MSAN blends with different PVC DP

The miscibility of P1/ α MSAN blends with different PVC DP was investigated with DSC. The DSC results shown in Table VI indicate that the blends of P1/ α MSAN with different PVC DP have the single T_g behavior. It indicates that α MSAN is easily

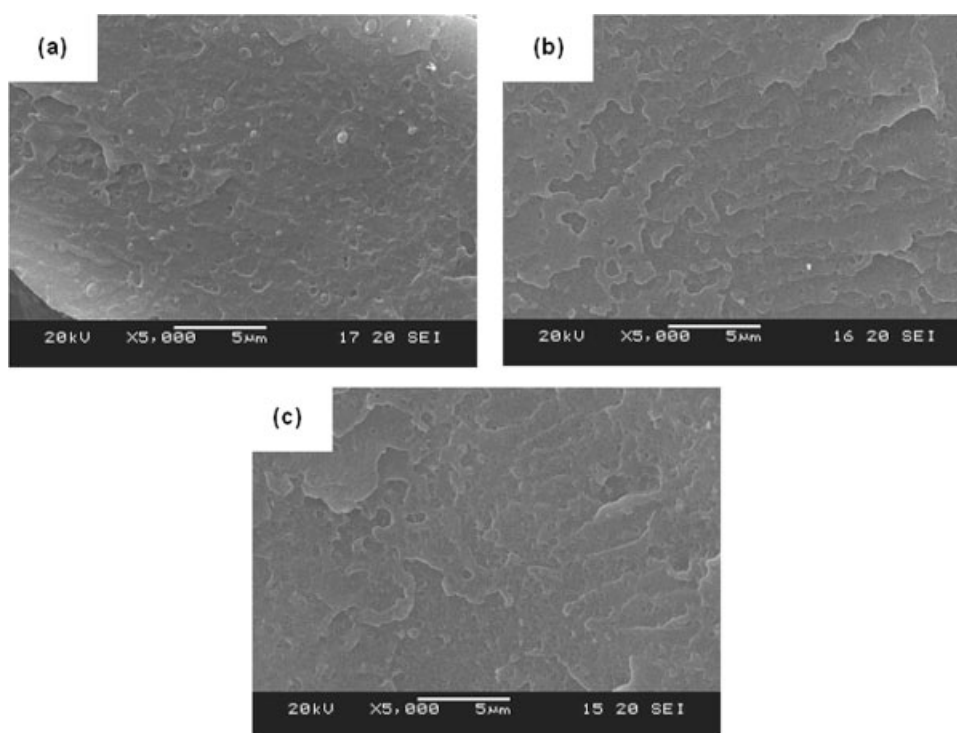


Figure 7 SEM images of cryogenically fractured surface of PVC and blends sample by compression molding; (a) P100/ α MSAN, (b) P130/ α MSAN, and (c) P170/ α MSAN.

mixed, even with high-DP PVC, because of the high interaction between PVC and α MSAN.

Figure 7 shows SEM images of a cryogenically fractured surface for P1 and P1/ α MSAN blends with different PVC DP. The SEM samples were prepared by compression molding. The P1 has a clear-cut surface, and nongelling parts appear as small and round particles. Compared to P1, the fracture behaviors of the P1/ α MSAN blends show different characteristics. All the P1/ α MSAN blends with changes in the PVC DP show clearer surface than those of the P1 indicating that α MSAN has very good miscibility with PVC, and subsequently, is miscible with PVC high-DP even when they blended with melt extrusion.

Mechanical properties of P1/ α MSAN blends with different DP of PVC

The equilibrium torques and degradation times of PVC (P1) and PVC/ α MSAN with different degree of polymerization of PVC (PVC DP) are shown in Figure 8. Compared to the P1 which is based on PVC DP 1000, the P1/ α MSAN blends show decreased equilibrium torque and an increased degradation time. PVC DP 1700/ α MSAN blend show the different trends to show a slightly higher equilibrium torque (16.5 Nm) and shorter degradation time (28 min) compared to the P1 (16 Nm, 30 min), but all these results would be considered to be in the possible processing condition. Therefore, it may be said that PVC high-DP/ α MSAN blends possess good processing properties enough to be studied for further improvements of the mechanical and physical properties of the blends.

The mechanical properties of P1/ α MSAN blends with changes in the PVC DP are shown in Table VII. As the DP of the PVC increases, all of three proper-

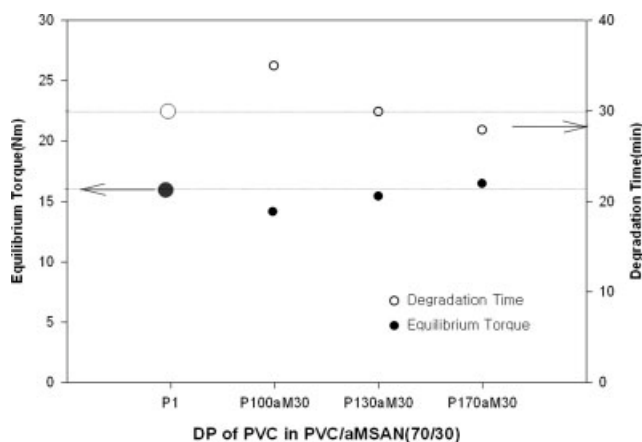


Figure 8 Equilibrium torque and degradation time of P1, α MSAN, and P1/ α MSAN blends with different PVC DP.

TABLE VII
Mechanical Properties of P1 and P1/ α MSAN Blends with Different Molecular Weight of PVC

Sample	Tensile strength (MPa)	Flexural strength (MPa)	Flexural modulus (MPa)
P1	85.3	112.7	2842
P100aM30	93.6	122.5	2920
P130aM30	97.0	138.2	3381
P170aM30	100.9	142.1	3577

For each test, three specimens were tested and the average value was obtained.

ties such as the tensile strength, flexural strength, and flexural modulus increase remarkably, indicating that the PVC high-DP/ α MSAN blend has good possibility for better products.

CONCLUSIONS

Because of the strong interaction between PVC and α MSAN, an α MSAN copolymer with 31 wt % AN was miscible with PVC over wide blend ratios, even when blended by melt extrusion. The resulting PVC/ α MSAN blends showed good mechanical and physical properties without any adverse effects. α MSAN copolymer with AN contents of 31 wt % was also miscible with high molecular weight PVC to result in enhance mechanical and physical properties. We believe that PVC with high molecular weight can be easily blended with α MSAN by an industrial extruder and thus, the mechanical, physical, and thermal properties of the PVC/ α MSAN blends can be improved.

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