

Effects of Composition and Temperature on Extrudate Characteristics, Morphology, and Tensile Properties of Acrylic Rubber–Blended PVC

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ABSTRACT: Poly(vinyl chloride) was blended with acrylic rubber over a range of compositions (5–40 wt % of the rubber), using a twin-screw extruder. Morphological properties of the blends were investigated as a function of rubber content and blending temperature, using a scanning electron microscopy. The mechanical properties of the blends were determined by a tensile test. Smooth extrudates were obtained at the blending temperature of 155°C. At a higher blending temperature (195°C), greater die swell ratio and/or melt-fractured extrudates were observed, depending on the rubber content. Miscible blends were obtained at low rubber contents (5–10 wt %). A dispersed particle morphology was observed from the extrudates containing the rubber content of 20–40 wt %, at 195°C. The ultimate tensile stress (UTS) and modulus of the blends decreased with the rubber content. The maximum tensile toughness was obtained for the blend with a rubber content of 20%, at a blending temperature of 155°C. © 2001 John Wiley & Sons, Inc. *J Appl Polym Sci* 80: 2523–2534, 2001

Key words: poly(vinyl chloride); acrylic rubber; blend; toughness; miscibility

INTRODUCTION

Poly(vinyl chloride) (PVC) is basically a rigid thermoplastic. It is inherently ductile because of the polarity and low-temperature molecular relaxation. However, it is brittle at high deformation rate and under concentrated stress, and the PVC is known as a notch-sensitive polymer.¹ PVC particles are rather difficult to fuse, requiring a high processing temperature. On the other hand, they have a very poor heat resistance, accompanied by a dehydrochlorination. The combination of difficult fusion and facile decomposition results

in a narrow range of acceptable processing temperature.

PVC powder is usually compounded with additives and/or blended with other polymers to improve properties such as impact strength (toughness), flexibility, processability, and heat resistance. For instance, impact strengths of PVC are improved by blending with various polymers such as chlorinated polyethylene (CPE),^{2,3} epoxidized natural rubber (ENR),^{4–8} nitrile rubber (NBR),^{9,10} methyl acrylate–butadiene–styrene (MBS) copolymer,^{11,12} acrylonitrile–butadiene–styrene (ABS) copolymer,¹³ and acrylate–methylacrylate copolymer.¹⁴ The efficiency of CPE in acting as an impact modifier depends largely on the chlorine content and distribution of the chlorine atoms. The CPE containing 25 to 40% chlorine, with a random distribution of chlorine atoms, was the best impact modifier

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for PVC. They have sufficient compatibility for adhesion to the PVC matrix without losing particulates and dispersibility. The polymers containing less than about 25% chlorine are incompatible with PVC and do not confer any property enhancement. The polymer with more than about 48% are highly compatible with PVC and become solubilized (miscible). In this case, CPE acts as a plasticizer rather than as an impact modifier.

PVC/ENR blend was studied extensively by Ishiaku et al.^{4–8} It was found that the two polymers were miscible, as shown by having a single tangent peak in a dynamic mechanical thermal analysis (DMTA) thermogram. The position of the peak, however, changed with the composition, which was in good agreement with the Gordon–Taylor equation.¹⁵ Results from Fourier transform infrared spectroscopy (FTIR) also revealed that hydrogen bonding, which was extensively involved in the blend system, is responsible for the miscibility of this polymer pair. Even though PVC and ENR were miscible, the impact strength of the PVC was improved, reaching a maximum value at the ENR content ranging between 7 to 9 wt %. A scanning electron micrograph of the etched specimen revealed some trace of the dispersed rubber particles at the submicron level. Ishiaku et al.^{4–8} explained that the higher impact strength in this case was attributed to the retention of some of the rubber microgel phase, which was finely dispersed in the PVC matrix.

Another well-known example of PVC-based polymer blend is NBR/PVC blend. This blend can be either miscible or immiscible (but compatible), depending on the composition and acrylonitrile content (% AN). At a PVC content of greater than 50%, an immiscible blend was obtained.⁹ Properties of this blend were predominated by PVC phase. The rubber serves as an impact modifier and/or a nonmigratory plasticizer. On the other hand, ozone resistance of NBR can be improved by incorporating about 10–15% of PVC. The resulting blend is used as an electrical insulator. In addition to the effect of composition, morphology of the blend is also affected by acrylonitrile (AN) content. Matsuo¹⁰ observed very fine rubber particles (~ 0.1 – $0.5 \mu\text{m}$ in diameter) in NBR/PVC (100/15) blend, having an AN content of 20%. However, as the acrylonitrile content was increased to 40%, the phase boundaries entirely destroyed and the rubber particle size decreased to below 10 nm in diameter.

This work concerns blending of PVC with acrylic rubber (AR). The acrylic rubber was con-

sidered here for the three following reasons: (1) the outstanding properties of the rubber, (2) the possible compatibility between the rubber and PVC, and (3) the commercial availability of the rubber. Acrylic rubber is characteristically superior in terms of elasticity, heat resistance, oil resistance, and processability (having low viscosity),¹⁶ and has been used as gaskets, hoses, seals, O-rings, and adhesives. By blending these two polymers, PVC products with improved toughness and/or heat resistant are expected, depending on the state of miscibility and the morphology. In this work, it was presumed that the polarity of an ester group in the acrylic rubber is sufficiently strong so that the interfacial adhesion (compatibility) between PVC and AR is good. The means that acrylic rubber can be used for blending with PVC, without employing any compatibilizing methods. The aim of this study is to examine morphological and mechanical properties of the acrylic rubber–blended PVC, as a function of rubber content and blending temperature.

EXPERIMENTAL

Raw Materials

PVC compound (B0303 CLA, suspension grade) was supplied by Thai Plastic and Chemicals Public Co. (Thailand). Acrylic rubber (Grade AR71) was obtained from Zeon Advanced Polymix Co. (Thailand). The major component of the acrylic rubber was poly(ethyl acrylate) (PEA), which contained a minor amount ($\sim 5\%$ bw) of chlorine cure-site monomer.

Mixing and Blending

The PVC and AR were blended at various compositions [95/5, 90/10, 80/20, 70/30, and 60/40 (PVC/AR % w/w)]. Prior to a melt blending, the polymers were dry-blended using a spinner for 3 min. After that, a twin-screw extruder (Haake PolyLab-rheomex CTW 100P) was used to prepare the blends. Two different temperature profiles were used in this work, as shown in Table I. In this study, the temperature at the die zone was referred to as blending temperature.

The blending process was started by loading a required amount of the dry blend into the hopper. The screw-rotating speed was 80 rpm. The compound was then extruded through a die. The die used was a three-strand die (each strand having a

Table I Temperature Profiles in the Extruder

Zone	Zone 1 (near hopper)	Zone 2 (middle)	Zone 3 (near die)	Die Zone
Temperature profile 1 (°C)	140	145	150	155
Temperature profile 2 (°C)	180	185	190	195

diameter of 3 mm), thus producing three extrudates at the die exit. The extrudates were then passed through a water bath by the use of a pull-off unit before undergoing the pelletization unit to produce granules. The granules were then repassed through the extruder to produce a more homogeneous blend for further use.

Die Swell Measurement

The die swell of the extrudate was directly determined by calculating the ratio of the diameter of the extrudate (D_{ext}) to that of the die (D_{die}), as shown in the following equation:

$$\text{Die swell ratio} = D_{\text{ext}}/D_{\text{die}}$$

It should be noted that the extrudate swell measurement in this work was based on the determination of the size of the extrudate diameter in the fully swollen extrudate, the technique of which was previously described.¹⁷

Test-Piece Fabrication

To investigate the mechanical (tensile) properties of the blend, dumbbell-shaped samples [in accordance with BS 2782 (Part 3, Method 320B)] were prepared using an injection-molding machine (Elite, E-80, Elite, Italy). The injection conditions used were as follows:

- Temperature from barrel to nozzle (°C) 170, 180, 190
- Injection speed (%) 99 (100% = 111 cm³/s)
- Mold temperature (°C) 25
- Cooling time (s) 10

The samples produced were kept under a controlled temperature of 25°C and 50% relative humidity for 24 h prior to testing.

Mechanical Properties

Mechanical properties of the blend were determined by tensile tests [in accordance with a standard test method (BS 2782)], using an initial

gauge length of 8 cm. Tensile measurements were carried out using an AGS-500D tensile-testing machine (Shimadzu; Japan). The cross-head speed used was 100 mm/min. Five specimens were tested for each blend and the averaged value was reported. Tensile toughness of various blends was determined by integration of the area under the obtained force-displacement traces.¹⁸

Scanning Electron Microscopy (SEM)

The extrudates and/or tensile specimens of PVC/AR blend were fractured after being frozen in liquid nitrogen. After that, the specimens were mounted on an aluminum stub and coated with carbon to reduce the charging effect.¹⁹ The carbon coating was performed using a vacuum-coating unit (JEOL JEE-400; JEOL, Peabody, MA) operated at 15 A. The SEM experiment was performed at a 15-kV accelerating voltage, using a JEOL (JSM-6301F) machine in conjunction with a back-scattering electron detector.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectra of various blends were obtained by using a Bio-Rad spectrometer (FTS 175; Bio-Rad, Richmond, CA). The sample was cast onto a ZnSe window cell, and the spectrum was recorded.

RESULTS AND DISCUSSION

Characterizations

Figure 1 shows FTIR spectra of PVC/acrylic rubber blends (90/10 and 60/40 wt %), at the blending temperature of 155°C. The FTIR spectra of the blends exhibited characteristic absorption peaks of both polymers. These include the peaks at 1732 cm⁻¹ (assigned to C=O of acrylic rubber), 1434 cm⁻¹ (assigned to δCH_2 of PVC), 1254 cm⁻¹ (assigned to δCH of PVC), 1163 cm⁻¹ (assigned to $\nu\text{C—O}$ of acrylic rubber), and 965 cm⁻¹ (assigned to νCH_2 of PVC). The absorption peaks at 614 and 695 cm⁻¹ represented $\nu\text{C—Cl}$ bonds of PVC (syn-

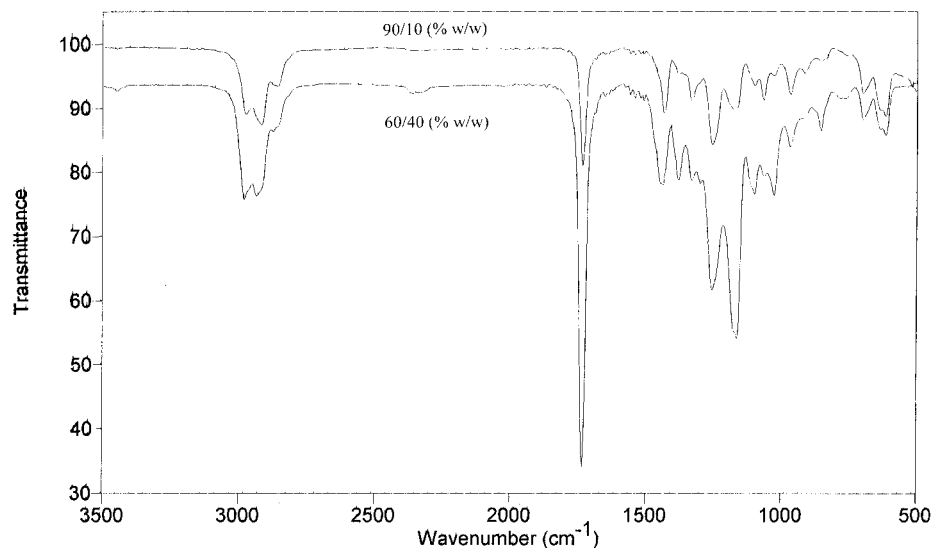


Figure 1 FTIR spectra of acrylic rubber-blended PVC.

diotactic and isotactic structures, respectively).²⁰ The position of the C=O peak remained constant, regardless of the rubber content and blending temperature, which suggested that an interaction between PVC and acrylic rubber in this study was insufficiently strong to perturb the local environment of the oscillating carbonyl bond and to shift the peak toward a lower wave number. In some polymer blends, however, a small shift ($<10\text{ cm}^{-1}$) in the carbonyl peak toward a lower wave number was used as evidence of miscibility, including polycaprolactone (PCL)/poly(vinylidene

chloride-co-acrylonitrile)²¹ and PVC/PCL²² blends, characterized by a strong hydrogen bonding between two phases.

Extrudate Characteristics

Extrudate characteristics (e.g., die swell, shark skin, melt fracture) deserve consideration, particularly in the manufacture of electrical wires, pipes, and sheets. Figure 2 shows the effect of rubber content and blending temperature on die swell ratio of various PVC/AR extrudates. It can

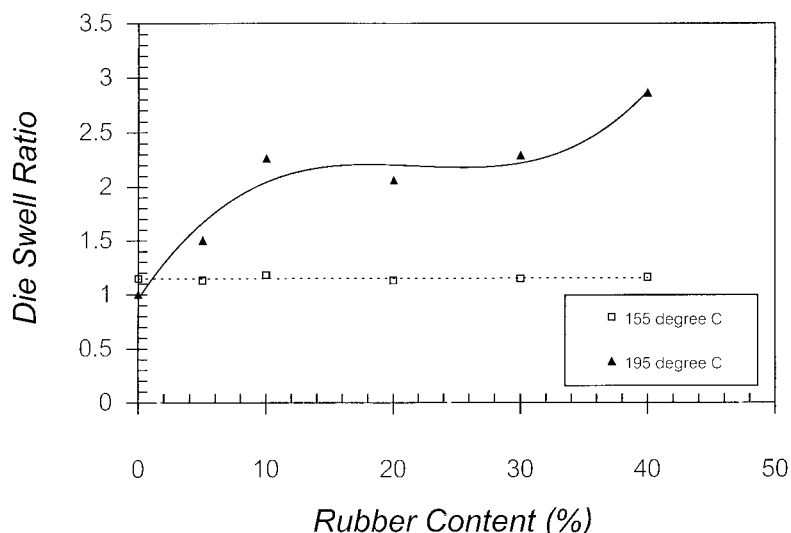


Figure 2 Relationships between rubber content, blending temperatures, and die swell ratios of the PVC/AR extrudates.

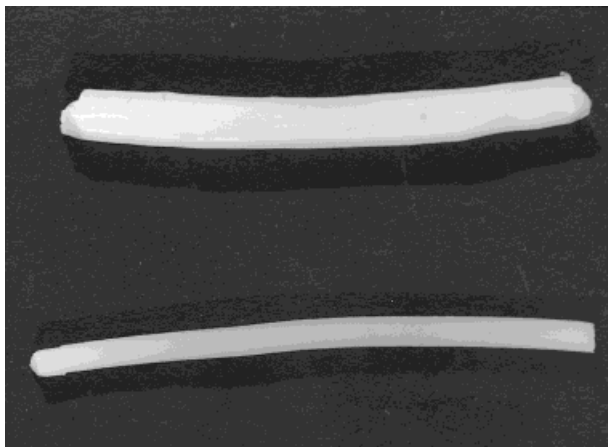


Figure 3 Photograph of PVC/AR extrudates (80/20% w/w) at 155°C (bottom) and 195°C (top).

be seen that, at lower temperature (155°C), smooth extrudates with die swell ratios of about 1.15 were observed, irrespective of the rubber content. As the temperature was increased to 195°C, die swell ratios of the extrudates, with 10–20% rubber content, increased to about 2.2 (see also Fig. 3). In general, die swell ratios of most polymer extrudates decreased with temperature²³ because the elastic characteristic reduces with temperature. However, Huneault et al.²⁴ noted that PVC behaved differently from other polymers, with respect to the change in die swell with temperature. According to Huneault, die swell ratios of PVC increased from 1.3 to 1.5 as the temperature was raised from 170 to 190°C, which was claimed to be associated with its morphology. By increasing the temperature, the degree of gelation (or fusion) of the PVC particles increased. As a result of the fusion, the entanglements and elasticities of the PVC increased, leading to a greater degree of swelling. Relative to this work, it may be possible that at 195°C the PVC phase in the blend experienced the fusion and thus the die swell ratio increased.

Figure 2 shows that the die swell ratio of pure PVC decreased with temperature (from 1.15 to 1). This trend is opposite to that of the acrylic rubber-blended PVC. In this regard, the presence of the rubber phase in the blend had to be taken into account. It was possible that the actual temperature of the blend was higher than that of pure PVC, resulting from more friction and frictional heat.²⁵ As a result, the fusion of PVC phase in the blend was accelerated. These results suggested that die swell ratios of PVC could be either decreased or increased with temperature, depend-

ing on the actual temperature of the molten PVC. This conclusion seemed to be supported by our observation noting that, at a higher extrusion screw speed (120 rpm), the die swell ratio of PVC was not decreased but tended to increase (from 1.2 to 1.3) as the temperature was raised from 170 to 200°C.

In addition to the die swell effect, melt-fracture characteristics were observed from the extrudates having higher rubber contents (30–40%), as shown in Figure 4. This flow instability (melt fracture) was also observed from the pure acrylic rubber extrudate. It seems that, at 195°C, the mixture in the barrel was basically an elastomer and thus the extrudate characteristic of the rubber (melt fracture) predominated the PVC phase. A similar behavior was also observed in the PVC/ENR blend (50/50% w/w).⁴

Morphologies of the Extrudates

Figure 5 shows scanning electron micrographs of various compositions of PVC/AR extrudates, at the blending temperature of 155°C. For the blend containing 10% AR, no phase separation can be observed [Fig. 5(a)], which suggested that the blend was miscible. Previous studies by Walsh et al.,^{26,27} using dynamic mechanical analysis to examine the miscibility between PVC and (meth)acrylate polymers, found that the two polymers can be either miscible or immiscible, depending on several factors: length of the ester side chain and the blending method. For PEA blended with PVC (at 50 : 50 weight ratio) via a solvent casting, a one-phase blend was observed. However, the PEA was immiscible with PVC prepared by *in situ* polymerization.

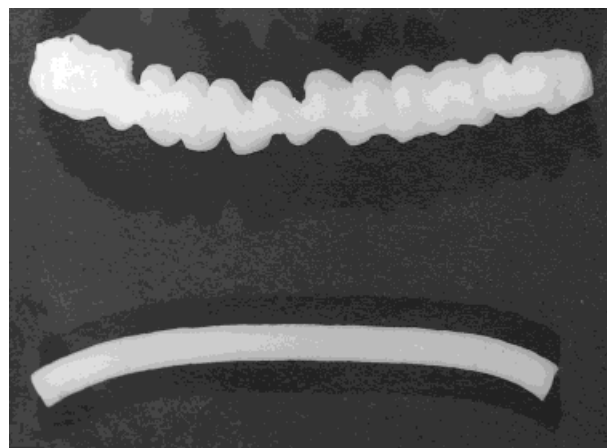


Figure 4 Photograph of PVC/AR extrudates (60/40% w/w) at 155°C (bottom) and 195°C (top).

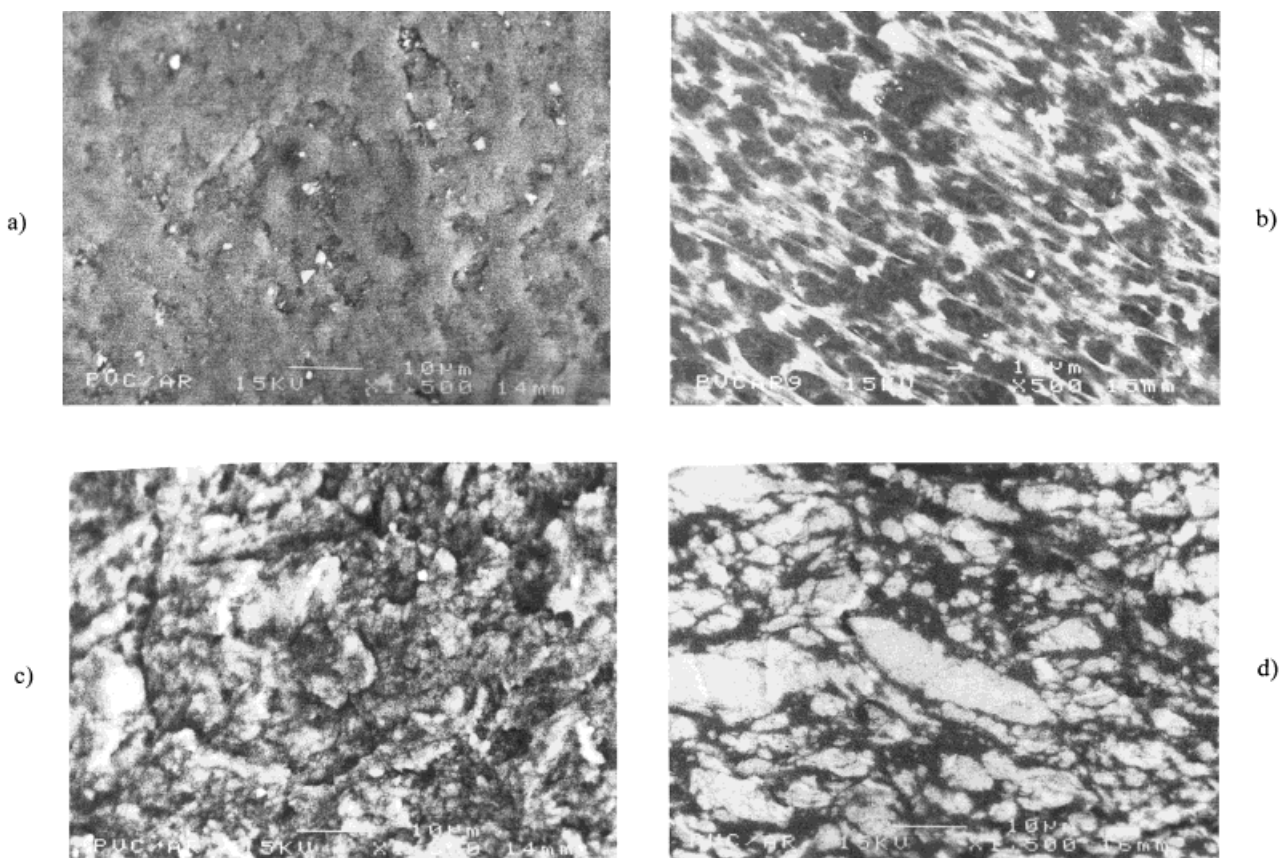


Figure 5 SEM micrographs of various PVC/AR extrudates: (a) 90/10% w/w; (b) 80/20% w/w; (c) 70/30% w/w; (d) 60/40% w/w. The blending temperature was 155°C.

As the rubber content was increased to 20%, phase separation can be seen clearly [Fig. 5(b)]. The dark phase represented the acrylic rubber, whereas the bright phase corresponded to the PVC, containing high atomic weight atoms (Cl). This morphology seemed to be a cocontinuous type. Increasing the rubber content to 30% revealed an interesting image [Fig. 5(c)], in which the two phases were not totally separate. Sub-inclusion of the rubber phase to the PVC phase can be seen. This was probably the result of a partial miscibility between the PVC and the acrylic rubber. At the rubber content of 40% [Fig. 5(d)] the PVC formed a discrete phase, whereas the rubber was more continuous, even through the latter was a lower weight fraction component. This was possibly because the rubber is softer than the PVC. Therefore, the rubber phase was deformed and dispersed throughout the system. This indicated that, at this condition, the viscosity ratio effect predominated the composition effect.

Figure 6 shows scanning electron micrographs of the PVC/AR extrudates, at a blending temperature of 195°C. The morphologies of these blends are significantly different from those at 155°C. The PVC formed a matrix phase, regardless of the rubber content. This effect was accompanied with a fusion of the PVC, as stated earlier. At the rubber content between 20 and 30%, typical dispersed particle morphology was observed. For the blend containing 20% rubber, the particle size ranged between 0.5 and 1 μm in diameter [Fig. 6(b)]. This particle size was rather high, compared to the optimum values for toughening PVC.²⁸ Finally, rubber particle size of the blend decreased [Fig. 6(d)] as the rubber content was raised to 40%.

Mechanical Properties

Figure 7 shows typical tensile force-displacement traces of the PVC/AR blends at various rubber contents, at a temperature of 155°C. The traces

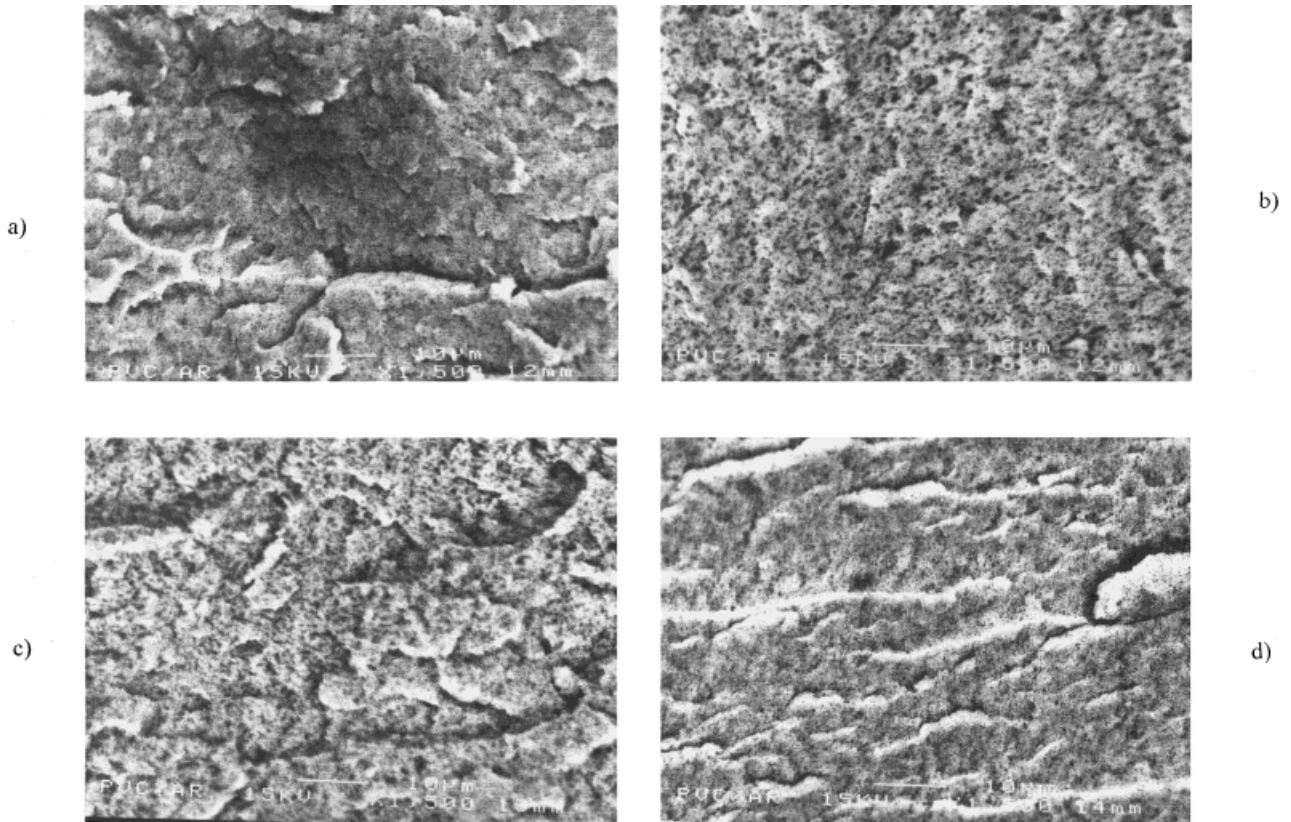


Figure 6 SEM micrographs of various PVC/AR extrudates: (a) 90/10% w/w; (b) 80/20% w/w; (c) 70/30% w/w; (d) 60/40% w/w. The blending temperature was 195°C.

significantly changed with rubber content. An increase in rubber content resulted in an increase in displacement accompanied by a decrease in ultimate tensile force. The effects of rubber content and blending temperatures on the modulus and ultimate tensile stress (UTS) of the blends are shown in Figures 8 and 9. Both parameters decreased with rubber content. The UTS was scarcely affected by blending temperature (Fig. 8). The modulus, however, significantly increased with temperature over the rubber content of 20–40% (Fig. 9). One possible explanation for this was the change in miscibility with temperature. In a miscible blend, the modulus may be slightly higher than a calculation-based rule of mixture.²⁹ It is a reduction in free volume, associated with the mixing process, that leads to a higher modulus. Previous work by Ishiaku et al.⁵ on PVC/ENR blends also showed that the relationship between low temperature storage modulus and composition of the blend positively deviated from the rule of mixture, but corresponded well to the Kleiner equation.³⁰ Similar results were also observed in

polystyrene (PS)/poly(2,6-dimethyl 1,4-phenylene oxide) (PPO) blend³¹ and PVC/NBR blend.³²

Figure 10 shows the changes in tensile toughness of PVC as a function of rubber content, for

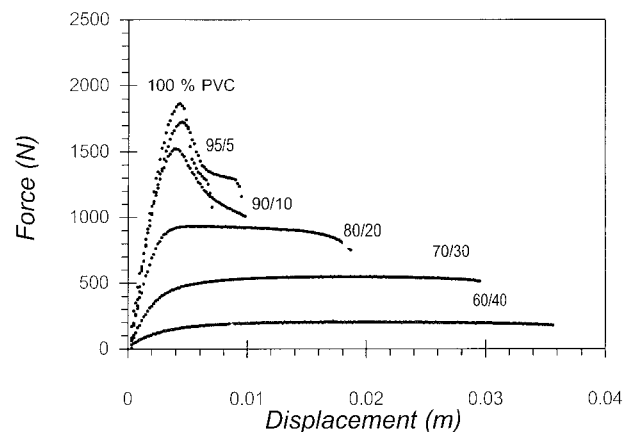


Figure 7 Force-displacement traces of PVC/AR blends at various compositions. The blending temperature was 155°C.

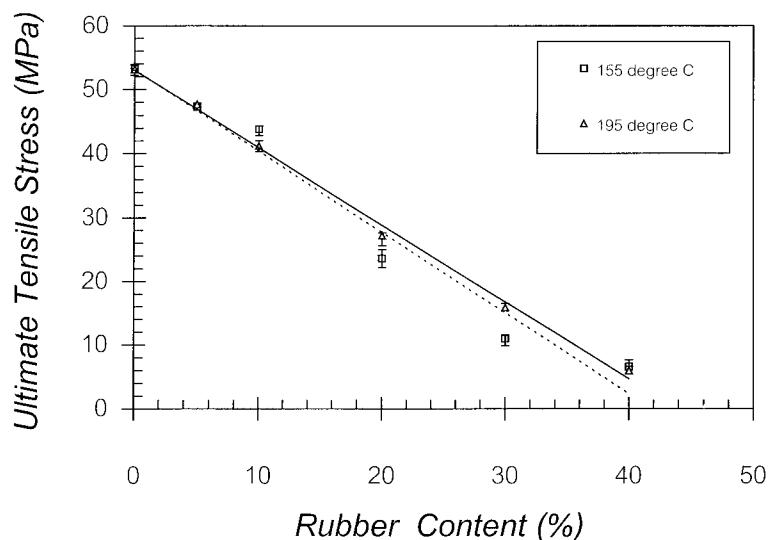


Figure 8 Ultimate tensile stress (UTS) of PVC/AR blend as a function of rubber content and temperature.

two different blending temperatures (155 and 195°C). It can be seen that the toughness did not increase linearly with the rubber content. At 155°C the toughness initially increased with rubber content up to the maximum value (18 nm). This maximum point corresponds to the rubber content of 20%. Beyond this rubber content, the toughness decreased, probably the result of a change in the morphology of the blend. This was similar to the results at 195°C blending temperature. The blend had the maximum toughness (14 nm) at the rubber content of 30%. At the rubber content of 10 and 20%, the toughness of the blend

at 195°C was lower than that at 155°C. However, at 30 and 40% rubber content, the grades of toughness between the two temperatures were very close.

Morphologies of the Injection-Molded Specimens

Figure 11 shows scanning electron micrographs of fractured surfaces of PVC/AR tensile specimens, at 155°C. At 5% rubber content, the blend was miscible, which was possibly one of the reasons why the UTS, modulus, and toughness of the blends were not changed with increasing temper-

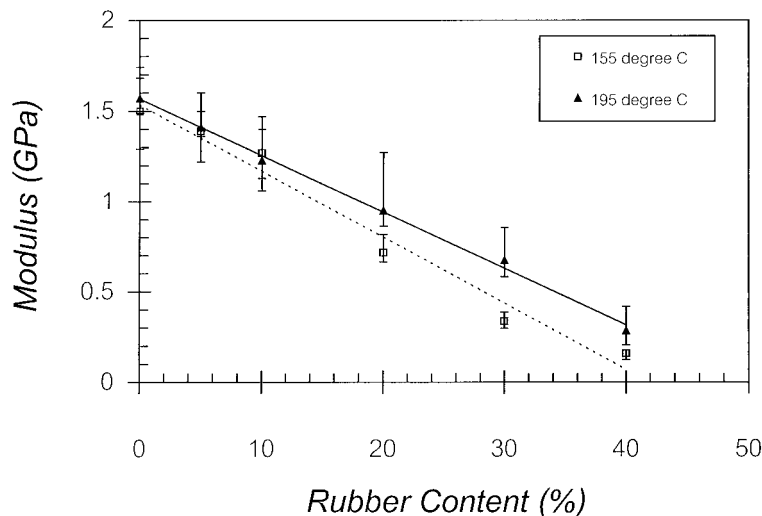


Figure 9 Modulus of PVC/AR blend as a function of rubber content and temperature.

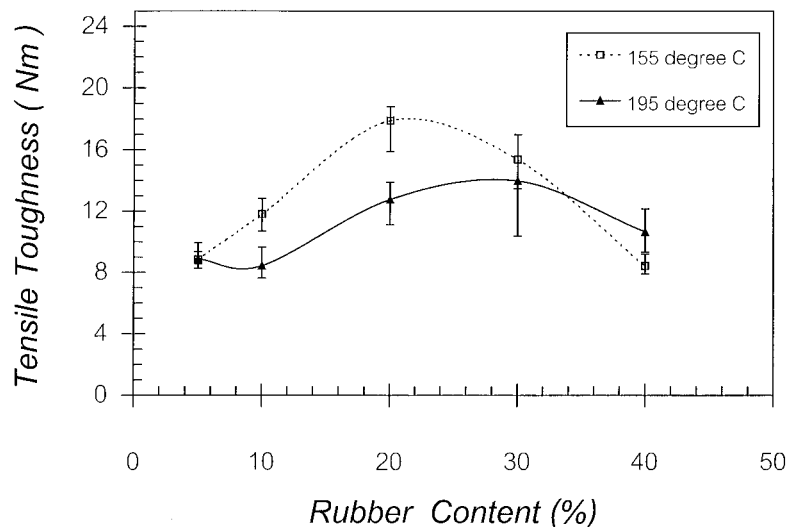


Figure 10 Tensile toughness of PVC/AR blend as a function of rubber content and temperature

ature. At a higher rubber content (20%), phase separation of the blend was observed, as shown by the dark discrete rubber phase [Fig. 11(a)], which is probably a reason why the maximum toughness was observed at this composition. According to a toughening mechanism,^{13,33,34} immiscibility between matrix and rubber is a strong prerequisite for the toughening effect. The rubber phase must be retained, in particle form, to induce a combination of craze and shear yielding.

Increasing the rubber content to 30 and 40% revealed two discrete domain phases [Fig. 11(b) and (c)], one of which was rich in the acrylic rubber and the other of which contained mainly the PVC domain with some rubber subincluded. The PVC and acrylic rubber were not totally phase separated but partially miscible at this condition, which could be the reason why the toughness of this blend decreased at the higher blending composition (30–40% AR). By comparing the scanning electron micrographs of the blends before and after injection molding, it was noteworthy that the morphologies were significantly changed. The PVC and the rubber phase were more miscible after the injection molding.

Figure 12 shows scanning electron micrographs of the PVC/AR specimens, at a blending temperature of 195°C. The morphologies of the blends containing high rubber contents (30 and 40%) at this temperature were different from those at 155°C [Fig. 11(b) and (c)]. Phase contrast between PVC and the acrylic rubber was less obvious as the temperature was raised to 195°C.

At this temperature, the PVC may have experienced the fusion. As a result, the PVC domains had lost their particulate nature and became more miscible with the rubber. This result was in good agreement with the mechanical test, showing that the modulus of the blends slightly increased with temperature.

By comparing the morphologies of the blends before and after injection molding, it was found that the dispersed particle morphology of the extrudates containing 20% AR [Fig. 6(b)] became less obvious after injection molding. The two phases tended to be more miscible. The specimen with a rubber content of 30% also retained the dispersed particle morphology [Fig. 12(b)], giving the maximum tensile toughness.

The above-noted results suggested that the dispersed particle morphologies of the blends might continue to change upon further processing. This effect may be unlikely in some other blends that have a very strong degree of immiscibility, such as PS/PMMA, PS/HDPE, and PC/PS blends.³⁵ However, for the PVC/AR blends, the longer the mixing, the more miscible the blend. This point is very important because, to achieve a higher toughness of the PVC, it is necessary to ensure that the dispersed particle morphology was retained in the final form of the blend.

Finally, it was worth considering the effect of screw-rotating speed on morphology of the blend. Figure 13 shows the morphology of PVC/AR (80/20%) extrudate, at a blending temperature of 155°C at 120 rpm. The rubber phase tended to be

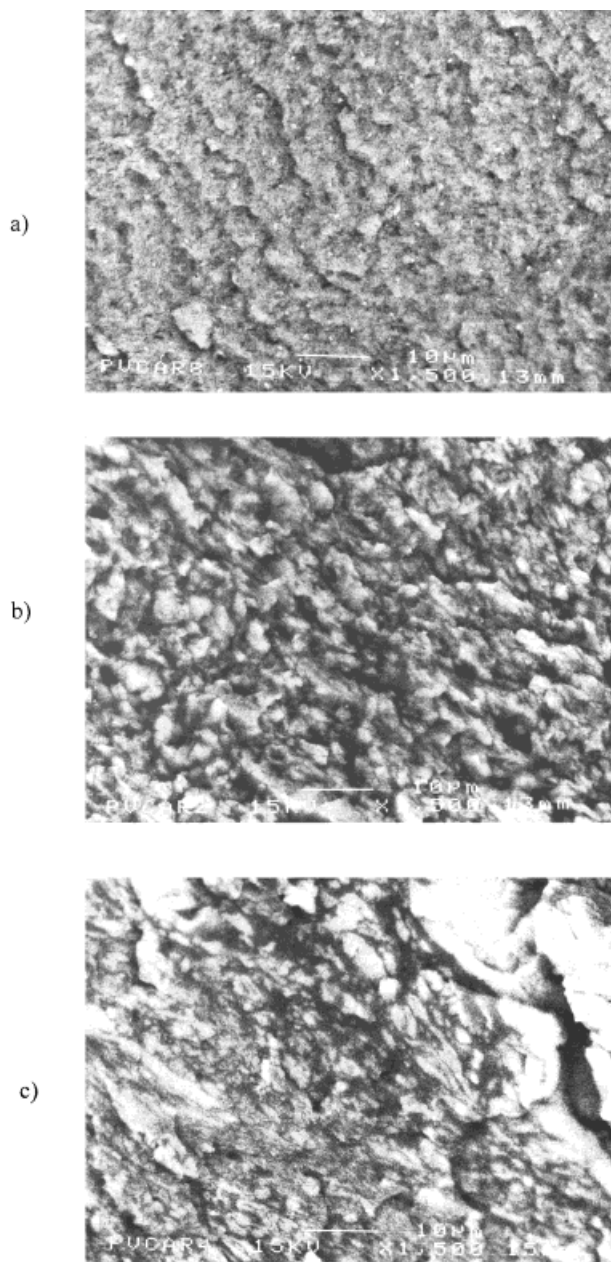


Figure 11 SEM micrographs of fractured surfaces of PVC/AR blends: (a) 80/20% w/w; (b) 70/30% w/w; (c) 60/40% w/w. The blending temperature was 155°C.

more continuous compared with that of the blend prepared at the screw speed of 80 rpm, for a given temperature [Fig. 5(b)]. As the blending temperature was raised to 195°C, well-dispersed particle morphology developed (Fig. 14). The rubber particle size of the blend prepared at 120 rpm ranged between 0.5 and 2 μm in diameter, which was higher than the particle size at 80 rpm. The differences may be attributed to a different polymer

melt residence time (in the extruder) between the two screw-rotating speeds. At 120 rpm, the polymer melt passed through the extrusion die faster and thus deformation and dispersion of the rubber phase were less intensive, and in turn led to the larger particle sizes. These results seem to support our comments that the PVC and acrylic rubber tended to be more miscible, if a sufficient mixing action was allowed. Further studies concerning the effect of screw-rotating speed on the

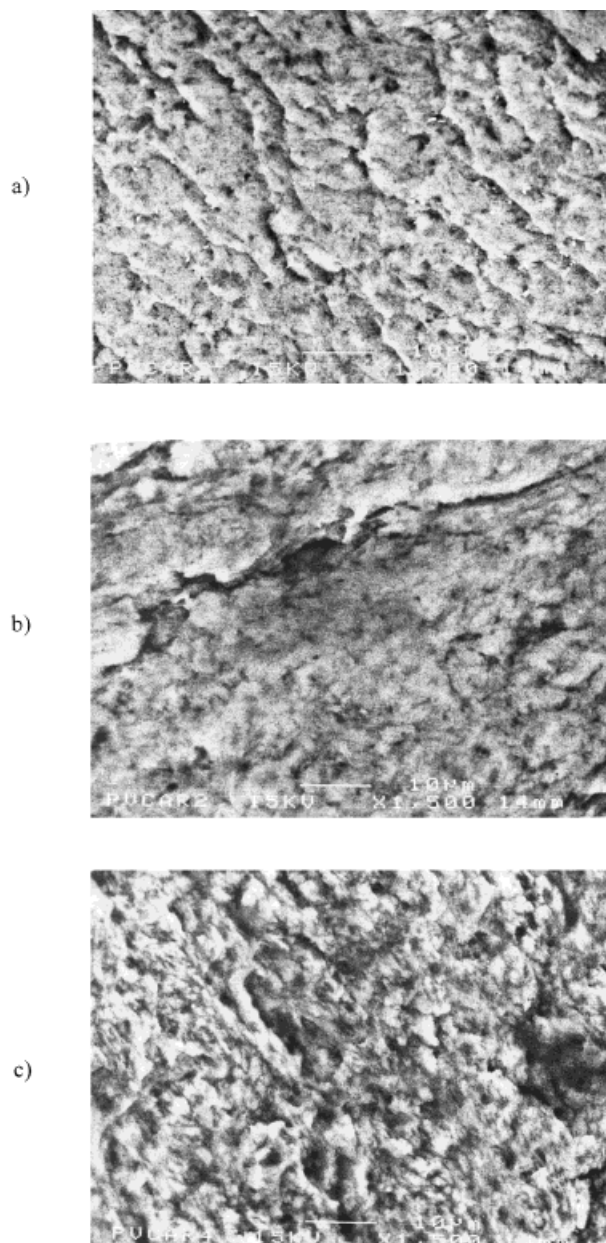


Figure 12 SEM micrographs of fractured surfaces of PVC/AR blends: (a) 80/20% w/w; (b) 70/30% w/w; (c) 60/40% w/w. The blending temperature was 195°C.

mechanical and morphological properties of the acrylic rubber-blended PVC are in progress.

CONCLUSIONS

The following conclusions can be drawn from this work:

1. One-phase PVC/acrylic rubber blends were observed at low rubber contents (5–10% by weight).
2. Acrylic rubber has no effects on die swell ratio of the PVC/AR extrudates, for a low blending temperature (155°C). At the higher temperature (195°C), the rubber content showed such effects. Die swell ratios of the blended PVC, with rubber contents of 10–20% at 195°C, were greater (87%) than those prepared at 155°C. At higher rubber content (30–40%), the extrudates exhibited melt-fracture characteristics.
3. The modulus and the ultimate tensile stress of the blends decreased with rubber content. At a rubber content ranging from 20 to 40%, the higher the temperature, the higher the modulus.
4. The optimum rubber contents, with respect to the maximum tensile toughness, were 20 and 30% by weight for the blending temperatures of 155 and 195°C, respectively.
5. At the blending temperature of 195°C, dispersed particle morphology can be ob-

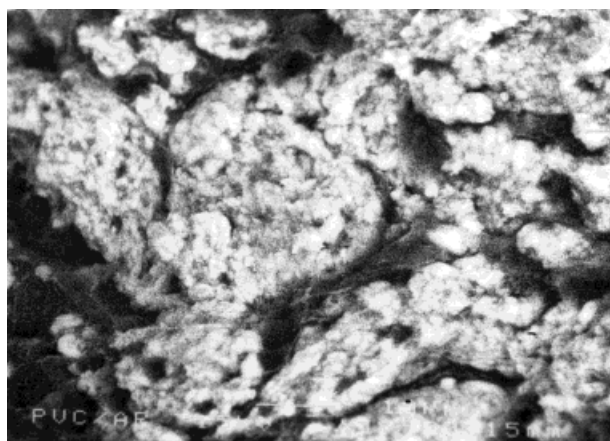


Figure 13 SEM micrographs of PVC/AR (80/20% w/w) extrudates at the blending temperature of 155°C at 120 rpm.

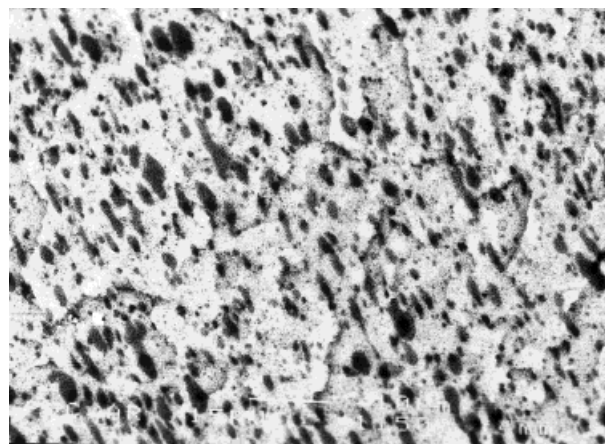


Figure 14 SEM micrographs of PVC/AR (80/20% w/w) extrudates at the blending temperature of 195°C at 120 rpm.

served from the extrudates, with rubber contents of 20 and 30%.

6. Morphological structures of the extrudates changed after injection molding, in which phase separation between PVC and the rubber particle became less obvious.

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