

# Effect of Acrylic Acid on the Properties of Recycled Poly(vinyl chloride)/Acrylonitrile–Butadiene Rubber Blends

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**ABSTRACT:** The effect of acrylic acid (AAc) on the torque, stabilization torque, mechanical energy, swelling behavior, mechanical properties, thermal stability, and morphological characteristics of recycled poly(vinyl chloride)/acrylonitrile–butadiene rubber (PVCr/NBR) blends was studied. The blends were melt mixed at a temperature of 150°C and rotor speed of 50 rpm. AAc was used to improve the compatibility of PVCr/NBR blends. Virgin PVCv/NBR blends were prepared to provide a comparison. It was found that PVCr/NBR + AAc blends exhibit higher stabilization torque, mechanical energy, stress at peak, and stress at 100% elongation, but lower elongation at break and swelling index

than those of PVCr/NBR and PVCv/NBR blends. SEM study of the tensile fracture surfaces of the blends indicated that the presence of AAc increased the interfacial interaction between PVCr and NBR phases, thus improving the compatibility between PVCr and NBR phases. However, thermal gravimetry analysis of the blends showed that the presence of AAc decreased the thermal stability of PVCr/NBR blends. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 96: 2181–2191, 2005

**Key words:** acrylic acid (AAc); poly(vinyl chloride) (PVC); acrylonitrile–butadiene rubber; morphology; mechanical properties

## INTRODUCTION

The blend of acrylonitrile–butadiene rubber (NBR)/poly(vinyl chloride) (PVC) is a miscible physical mixture of commercial importance. NBR can act as a permanent plasticizer for PVC applications such as in electrical wires and cable coatings, wrapping films for the food industry, conveyor belts, and domestic applications, to cite but a few primary examples. The presence of PVC helps to improve the ozone and ageing resistance of NBR.<sup>1,2</sup> It is well known that NBR has generally good resistance toward oil and low gas permeability. Its use in automotive applications is interesting, although the ageing resistance is limited because of the unsaturated backbone of the butadiene. Blending NBR with PVC enables this composite to be used in the automotive industry in such applications as gaskets, wires, and cable, and in the manufacture of sole, footwear, artificial leather, and the like.<sup>3–9</sup> The objective of blending plastic and rubber is the improvement of physical, thermal, and mechanical properties as well as the modification of processing characteristics and cost reduction of the final product.

However, some of these blends are incompatible. To improve the mechanical properties of these blends, it is

important to develop a proper control of phase morphology and good interfacial adhesion by using compatibilizing agents. Mathew and Thomas<sup>6</sup> reported the compatibilization of heterogeneous NBR/polystyrene (PS) blends by the addition of styrene–acrylonitrile copolymers, whereas Dahlan et al.<sup>7</sup> used liquid natural rubber (LNR) as a compatibilizer in NR/linear low-density polyethylene (LLDPE) blends. Sooppy et al.<sup>8</sup> found that for maleic anhydride–poly(propylene) (MAH–PP) compatibilized blends, the increase in tensile strength is attributed to the increased dipolar interaction between the MAH–PP and NBR phases, which causes an increase in interfacial adhesion between PP and NBR phases. In our previous work,<sup>9</sup> we reported the comparison of properties of virgin poly(vinyl chloride)/acrylonitrile butadiene rubber (PVCv/NBR) and recycled poly(vinyl chloride) (PVCr)/NBR blends. It was found that the PVCr/NBR blends exhibit lower properties (i.e., mechanical properties and swelling resistance) than those of PVCv/NBR blends. Here, we report the effect of acrylic acid (AAc) on processability, mechanical properties, swelling behavior, thermal stability, and morphology of PVCr/NBR blends.

## EXPERIMENTAL

### Materials

PVC powder [virgin PVC (PVCv), a suspension polymer with a *K* value of 65 and degree of polymerization

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TABLE I  
Inorganic Compound of Recycled PVC<sup>a</sup>

Composition	wt %	Composition	wt %
Al <sub>2</sub> O <sub>3</sub>	1.7	P <sub>2</sub> O <sub>5</sub>	0.025
MgO	0.39	SO <sub>3</sub>	0.15
SiO <sub>2</sub>	0.88	K <sub>2</sub> O	0.023
Cl	20	Fe <sub>2</sub> O <sub>3</sub>	0.052
CaO	13	CuO	0.19
TiO <sub>2</sub>	0.26	ZnO	0.086
Sb <sub>2</sub> O <sub>3</sub>	0.21	Br	0.036
PbO	1.2	SrO	0.013
Carbon	61	SnO <sub>2</sub>	0.029
Na <sub>2</sub> O	0.019		

<sup>a</sup> X-ray fluorescence spectrometer Rigaku RIX 3000.

of 920–1060] was supplied by Malayan Electro Chemical Industry (Penang, Malaysia). Recycled PVC (PVCr) from electrical wires and cables was supplied by Alfasya Jaya Sdn. Bhd. (Penang, Malaysia); the inorganic compound of recycled PVC is shown in Table I. NBR (34% acrylonitrile content) was obtained from Kumpulan Guthrie (Seremban, Malaysia). The dioctylphthalate (DOP), barium/cadmium (Ba/Cd) stearate, acrylic acid, and dicumyl peroxide (DCP) were obtained from Bayer (M) Sdn. Bhd. (Penang, Malaysia).

### Preparation of the blends

Formulations of the blends are given in Table II. Blend ratios of plastic and rubber were prepared at 80/20, 60/40, 50/50, 40/60, and 20/80 (wt/wt). The acrylic acid (AAc) concentration was fixed at 4 phr of plastic. The PVCr/NBR blends were prepared by melt-mixing in a Rheomix Polydrive R 600/610 apparatus (Haake, Bersdorff, Germany) at 150°C and rotor speed of 50 rpm. The amount of DOP and Ba/Cd stearate used for the mixing of the blends were 20 and 3 phr, respectively, based on total PVCr content in all composi-

TABLE II  
Formulations of PVCr/NBR Blends  
with Acrylic Acid (AAc)<sup>a</sup>

Material	Blend (phr)				
	1	2	3	4	5
PVCr	80	60	50	40	20
NBR	20	40	50	60	80
Stabilizer <sup>b</sup>	3	3	3	3	3
Plasticizer <sup>c</sup>	20	20	20	20	20
DCP <sup>d</sup>	0.2	0.2	0.2	0.2	0.2
Acrylic acid (AAc)	4	4	4	4	4

<sup>a</sup> A similar series of PVCr/NBR and PVCv/NBR blends were prepared without AAc.

<sup>b</sup> Ba/Cd stearate.

<sup>c</sup> Dioctylphthalate (DOP).

<sup>d</sup> DCP, dicumyl peroxide.

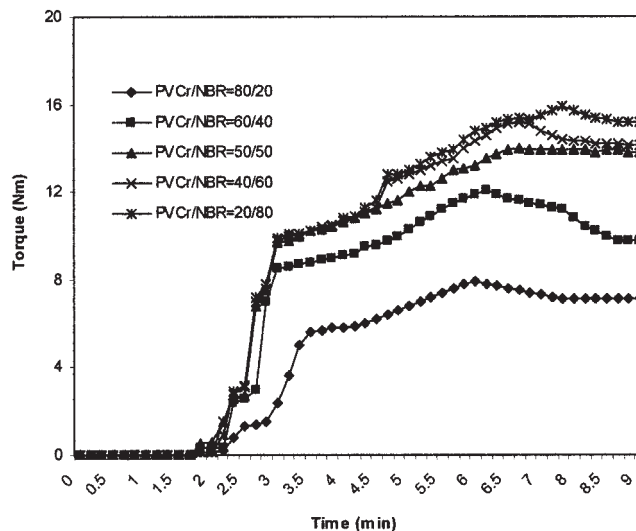


Figure 1 Torque versus time of PVCr/NBR + AAc with different blend compositions.

tions. The PVCr was initially premixed with stabilizer and plasticizer in a glass beaker for 5 min at room temperature. The PVCr with stabilizer and plasticizer, together with dicumyl peroxide and acrylic acid, was charged to the mixing chamber, allowed to equilibrate for 2 min, followed by the addition of NBR. Mixing was then continued until a constant torque was obtained. The total mixing time was 9 min. The compound was removed from the mixer and sheeted on a cold two-roll mill.

### Compression molding and testing

The samples of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends were compression molded in an electrically heated hydraulic press. Hot-press procedures involved preheating at 150°C for 4 min, followed by compressing for 4 min at the same temperature. Tensile tests were carried out with an M 500 model tensiometer (Testometric Co., Ltd., Rochdale, UK). Tests were done according to ASTM D412. Dumbbell-shape specimens (2 mm thickness) were cut from molded sheets with a S6/1/4.A model die cutter (Wallace Instruments, Kingston, UK). Five specimens were used in each case and the median value was selected. Reading of stress at peak, stress at 100% elongation ( $M_{100}$ ), and elongation at break ( $E_b$ ) were recorded directly from the digital displays at the end of each test.

### Swelling test

Determination of the swelling percentage of the blends was carried out according to ASTM D471. The test pieces of the blends (30 × 5 × 1.5 mm) were

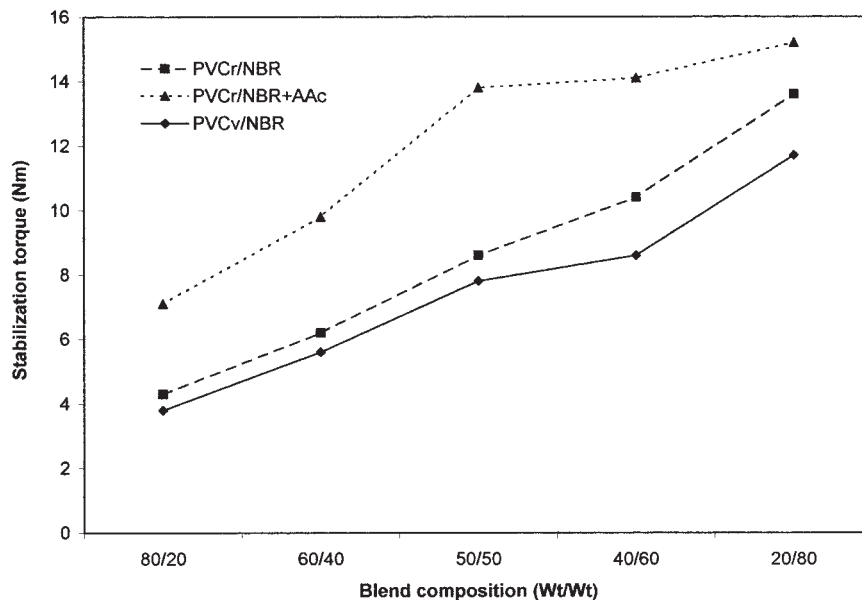


Figure 2 Stabilization torque versus blend composition of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends.

weighed using an electrical balance and this was considered to be original weight. The test pieces were immersed in toluene at room temperature for 46 h, after which were removed from the toluene, wiped with tissue paper to remove excess toluene from the surface, and weighed (swollen weight). A similar test procedure was used in IRM 903 oil for 70 h at room temperature. The swelling index of the blends was calculated as follows:

$$\text{Swelling index} = \frac{\text{swollen weight}}{\text{original weight}} \quad (1)$$

#### Scanning electron microscopy (SEM) study

Morphology studies of the fracture surfaces of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends were carried out using an S360 model scanning electron microscope (Leica, Cambridge, MA). Surfaces of the sample were coated with a thin gold layer of about 20 nm thickness.

#### Infrared spectroscopy study

The FTIR spectrum was obtained using a Perkin-Elmer 2000 series instrument (Perkin-Elmer Cetus In-

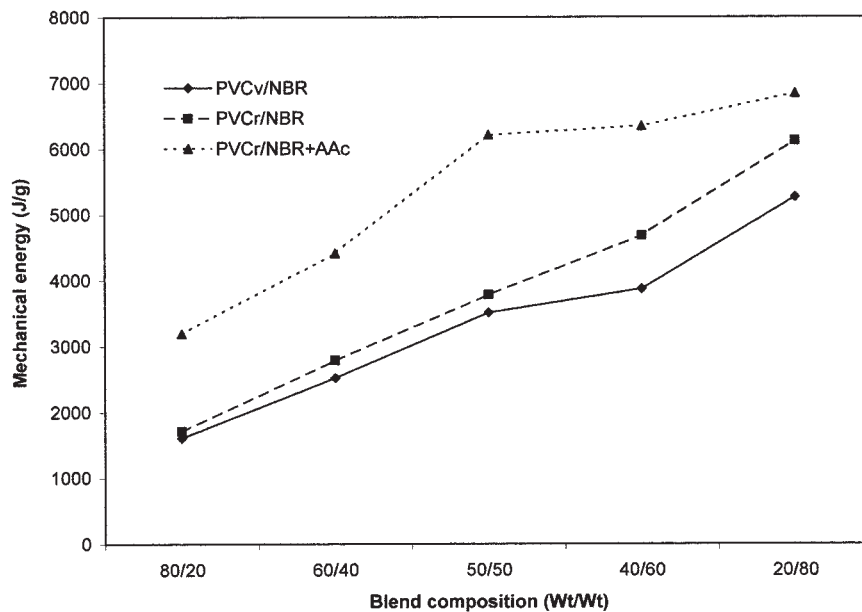


Figure 3 Mechanical energy versus blend composition of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends.

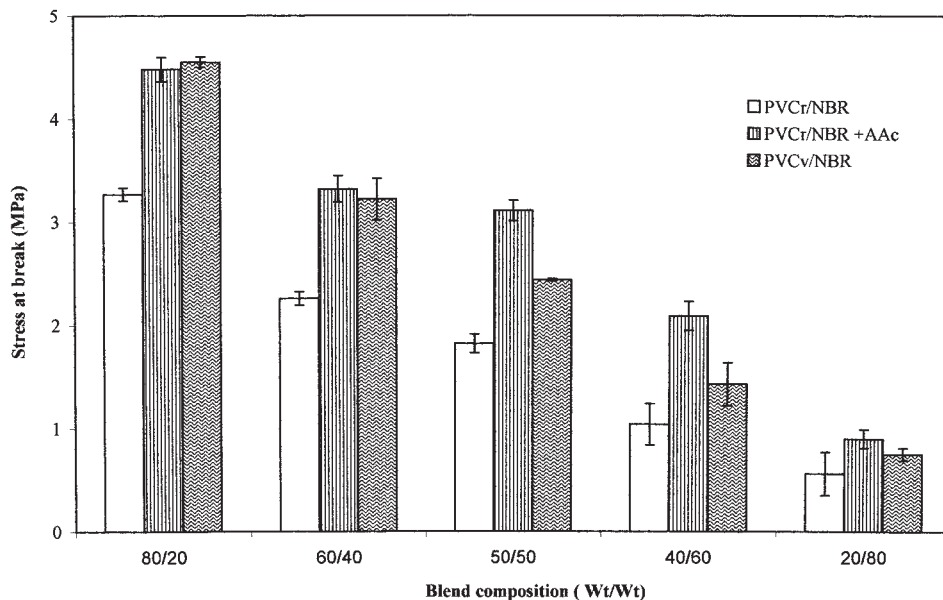


Figure 4 Stress at peak versus blend composition of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends.

struments, Norwalk, CT). The spectrum resolution was  $4\text{ cm}^{-1}$  and the scanning range was from  $600$  to  $4000\text{ cm}^{-1}$ . The samples for FTIR were first prepared by using an attenuated total reflectance (ATR) technique, subjected to FTIR analysis immediately afterward.

#### Thermogravimetric (TG) analysis

Thermal degradation analysis of the PVCv/NBR, PVCr/NBR, and PVCr/NBR + AAc blends was carried out using a Perkin-Elmer TGA-7 thermogravimetric analyzer. The weight of samples was about 10

mg. The weight change of the blends was investigated from room temperature to  $590^\circ\text{C}$ , at a heating rate of  $10^\circ\text{C}/\text{min}$  in a nitrogen atmosphere. The first derivative of the thermogravimetric curve was calculated using the in-built software.

## RESULTS AND DISCUSSION

#### Processing characteristics

The processing characteristics of the blends were investigated using Haake Rheomix plastographs. Figure

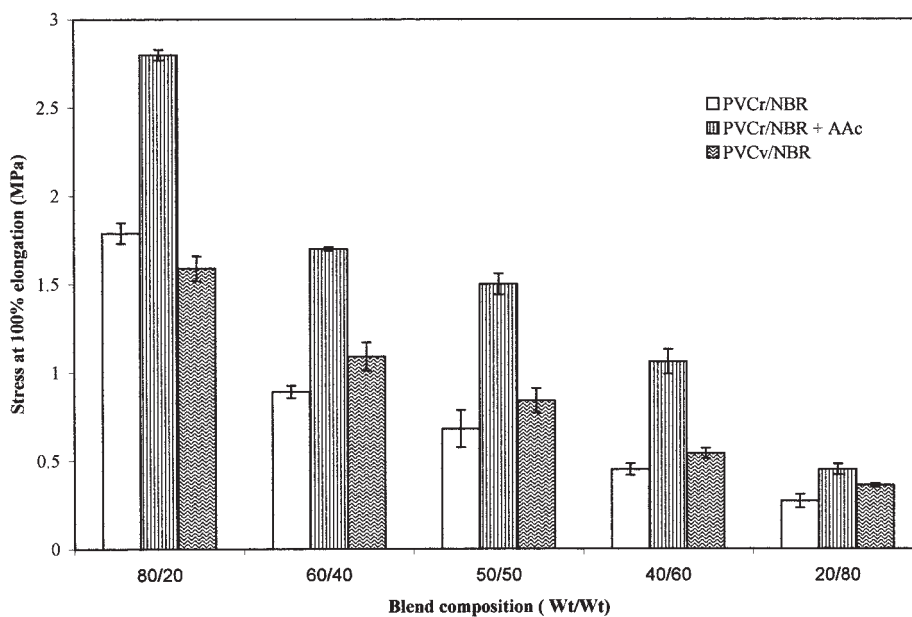
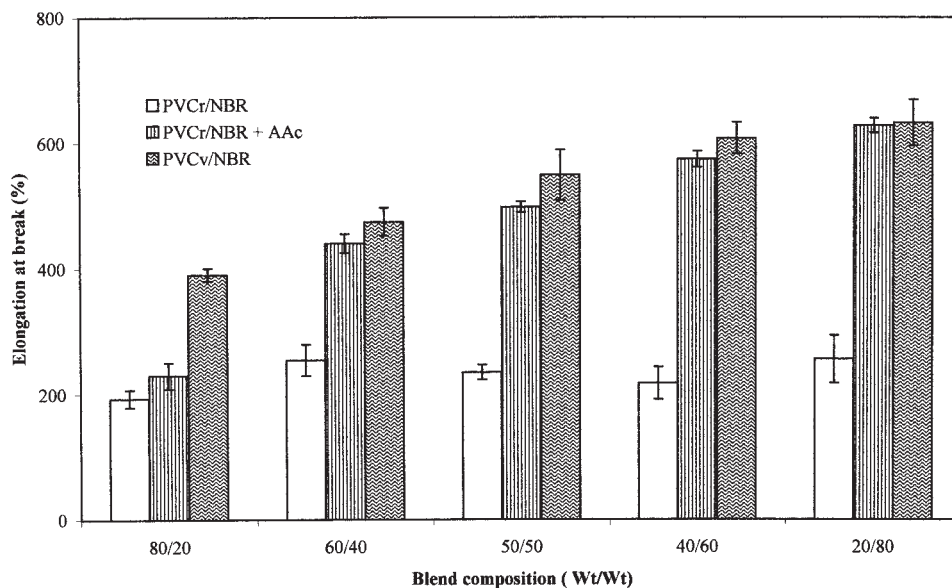


Figure 5 Stress at 100% elongation versus blend composition of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends.

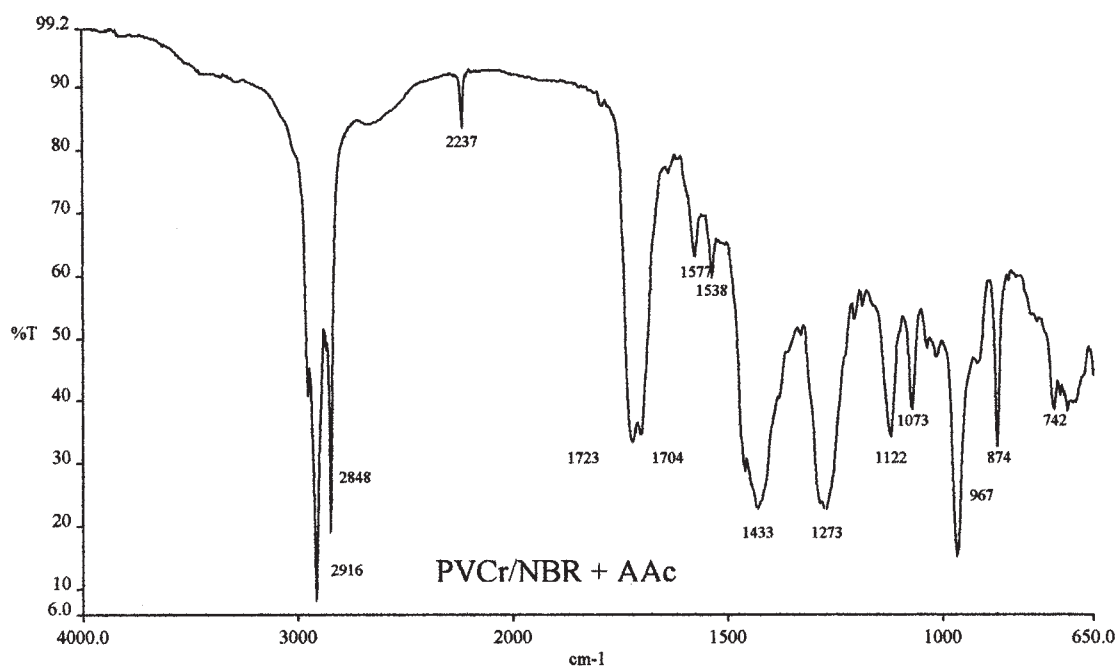


**Figure 6** Elongation at break versus blend composition of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends.

1 shows the torque–time relationship obtained from the Haake Rheomix for PVCr/NBR + AAc blends with different blend compositions. For PVCr/NBR + AAc blends, PVCr was charged to the mixing chamber together with dicumyl peroxide (DCP), acrylic acid, stabilizer, and plasticizer (DOP). The rotors were immediately started and the torque increased as a result of the resistance exerted on the rotor by the nonmolten recycled PVC. As the PVCr melted and was subjected to mechanical shearing,

the torque initially increased because of the acrylic acid-grafted PVCr, after which then the torque stabilized. The torque rose again sharply after NBR was charged into the mixing chamber at 2 min. As the NBR melted, the viscosity again decreased until the equilibrium torque was achieved when the blends become homogenized.

The stabilization torque at 9 min versus blend compositions of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends is shown in Figure 2. It can be seen



**Figure 7** FTIR spectrum of PVCr/NBR + AAc blend.

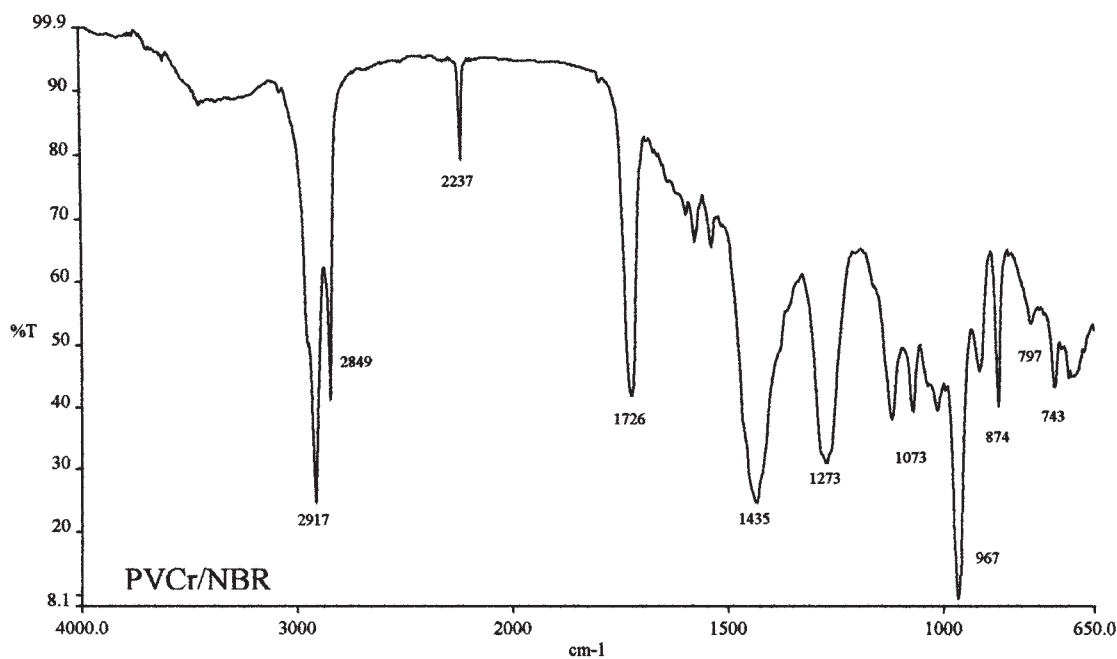


Figure 8 FTIR spectrum of PVCr/NBR blend.

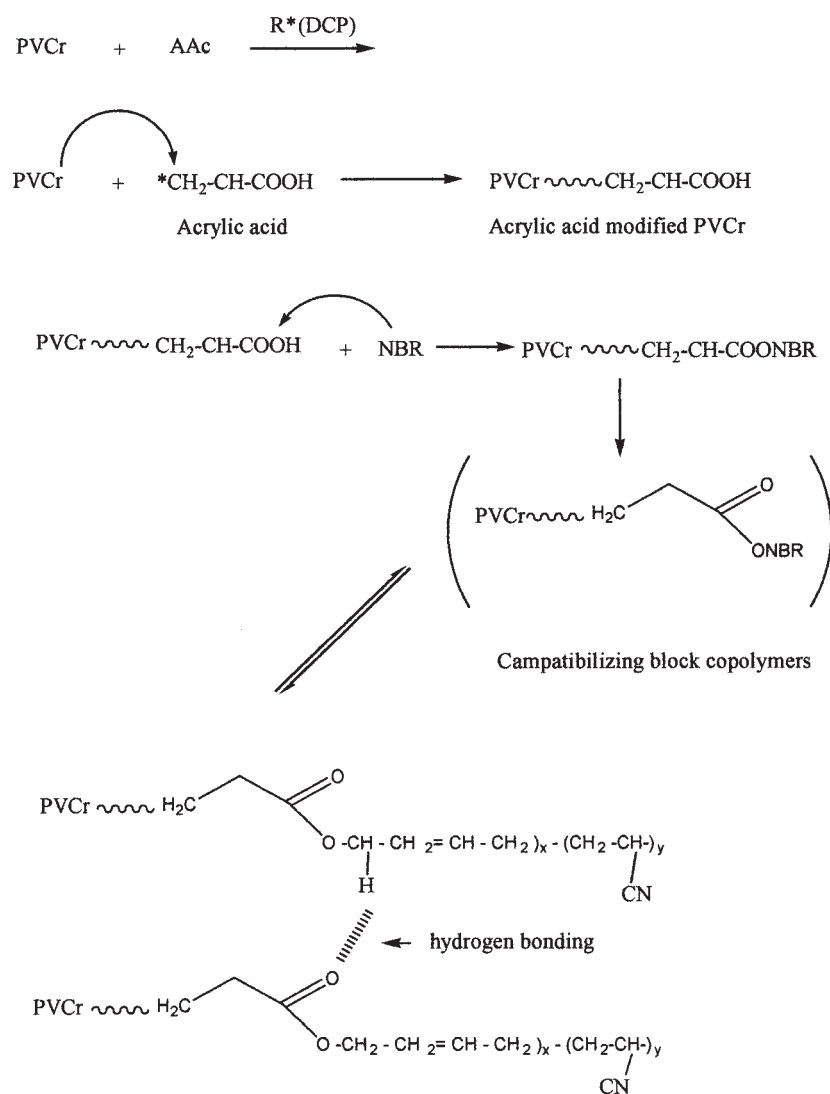
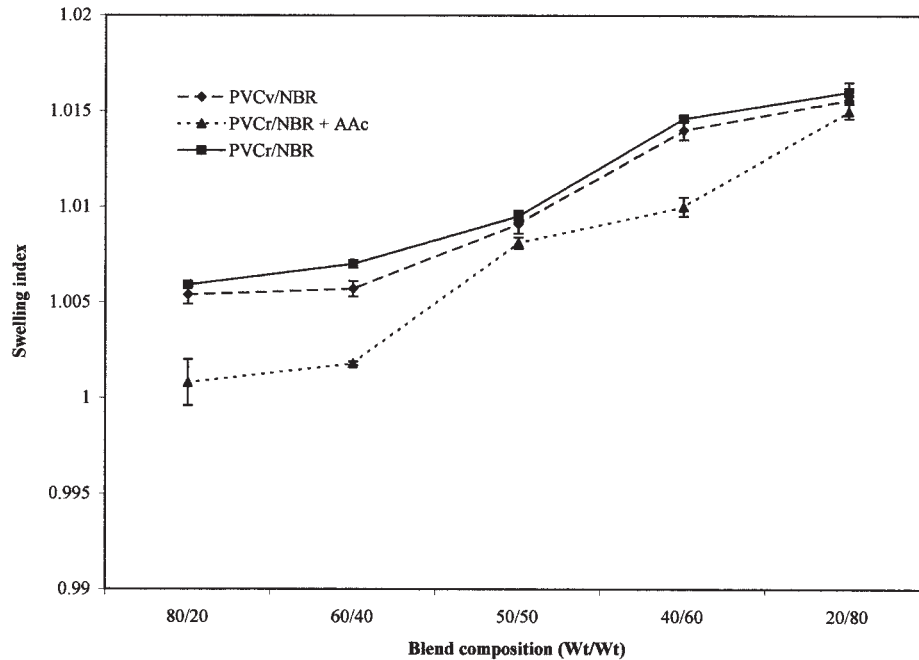


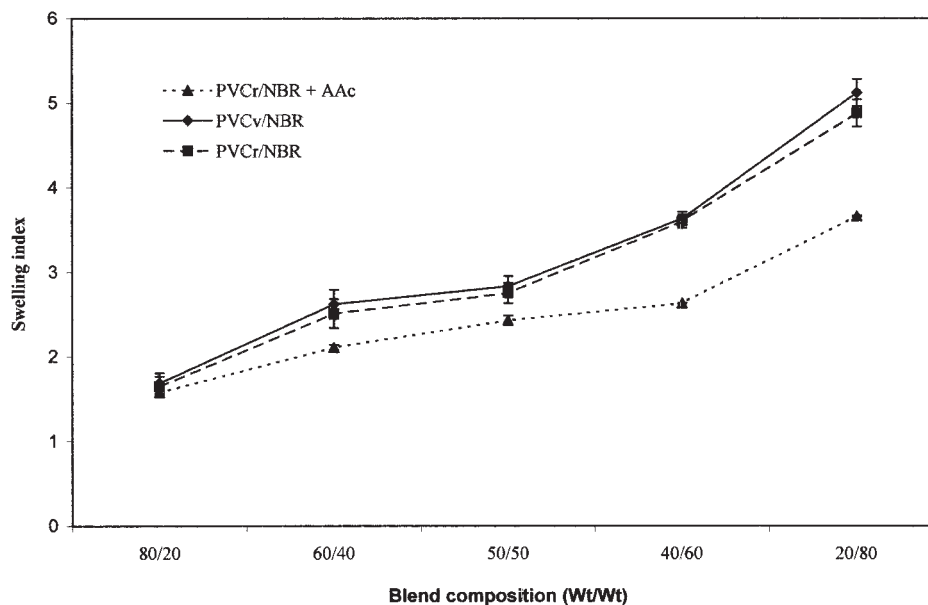
Figure 9 Reaction mechanism of acrylic acid grafted recycled PVC with NBR.



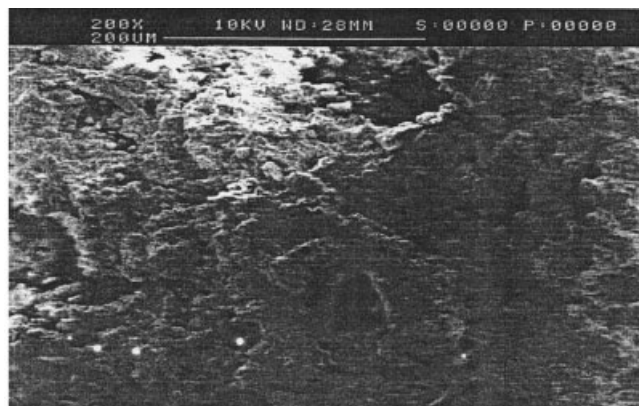
**Figure 10** Swelling index versus blend composition of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends that were immersed in IRM 903 oil for 70 h.

that for all blends the stabilization torque increases with increasing NBR content. As the NBR content in the blend increases, the blend viscosity is increased. The increase in viscosity, as a result of the incorporation of rubber, was previously reported by various researchers.<sup>10-14</sup> However, at a similar blend composition, PVCr/NBR + AAc blends exhibit higher stabilization torque than that of PVCr/NBR and PVCv/

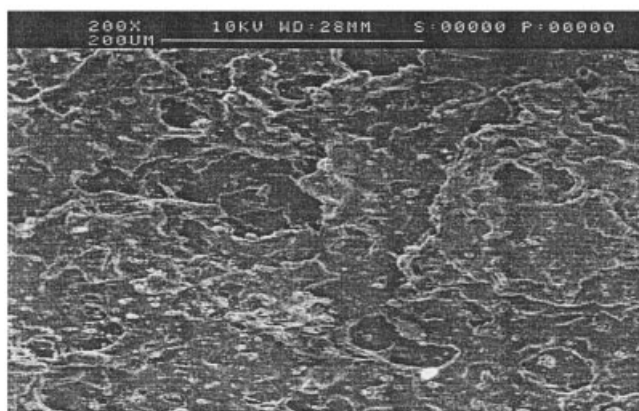
NBR blends. Given that stabilization torque is indicative of stable melt viscosity, it is clear from the above results that incorporation of AAc in the PVCr/NBR blends resulted in a higher energy requirement. In our previous work<sup>9</sup> we reported that the work done to process the blends ( $dE/dt$ ) is energy consumed during rotation and can be expressed as  $WM$ , where  $W$  is the torque and  $M$  is the angular velocity. Thus, an expres-



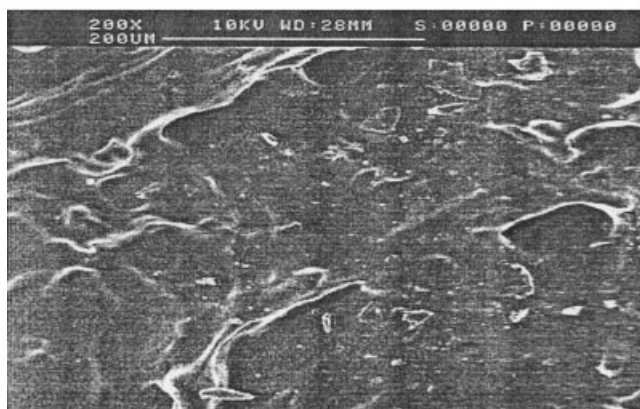
**Figure 11** Swelling index versus blend composition of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends that were immersed in toluene for 46 h.



a



b



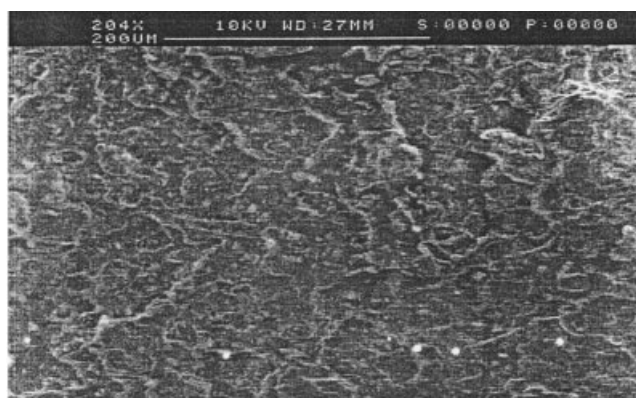
c

**Figure 12** SEM micrographs of tensile fracture surfaces of PVCr/NBR blends: (a) 80/20; (b) 50/50; (c) 20/80.

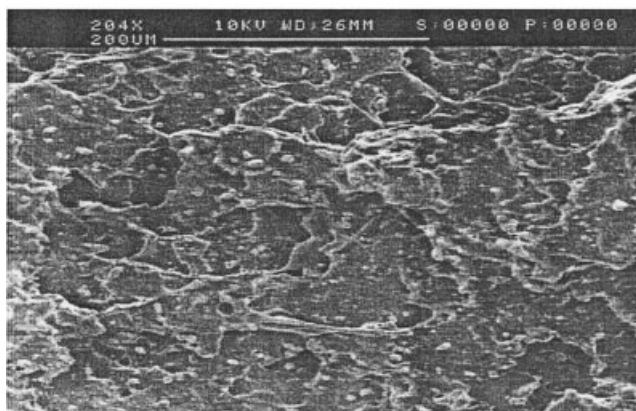
sion for the energy can be derived as  $dE = (WM) dt$ . By integration, the mechanical energy for processing the blends at a given temperature and rotor speed is obtained.

Figure 3 shows the effect of blend composition on mechanical energy of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends. It can be seen that the mechanical energy of all blends increases with increasing NBR content and at a similar blend composition,

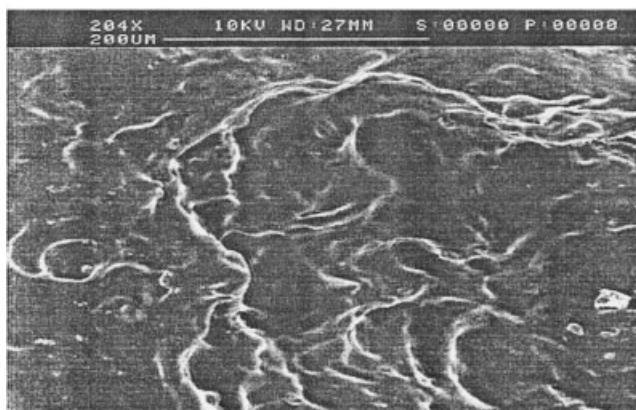
PVCr/NBR + AAc blends show the highest mechanical energy. Various researchers<sup>15–18</sup> reported the increase in viscosity on compatibilization of immiscible polymer blends. According to George et al.,<sup>19</sup> upon compatibilization of an immiscible blend, the compatibilizer will generally locate at the interface between the dispersed phase and the matrix, which leads to an increase in interfacial thickness.



a



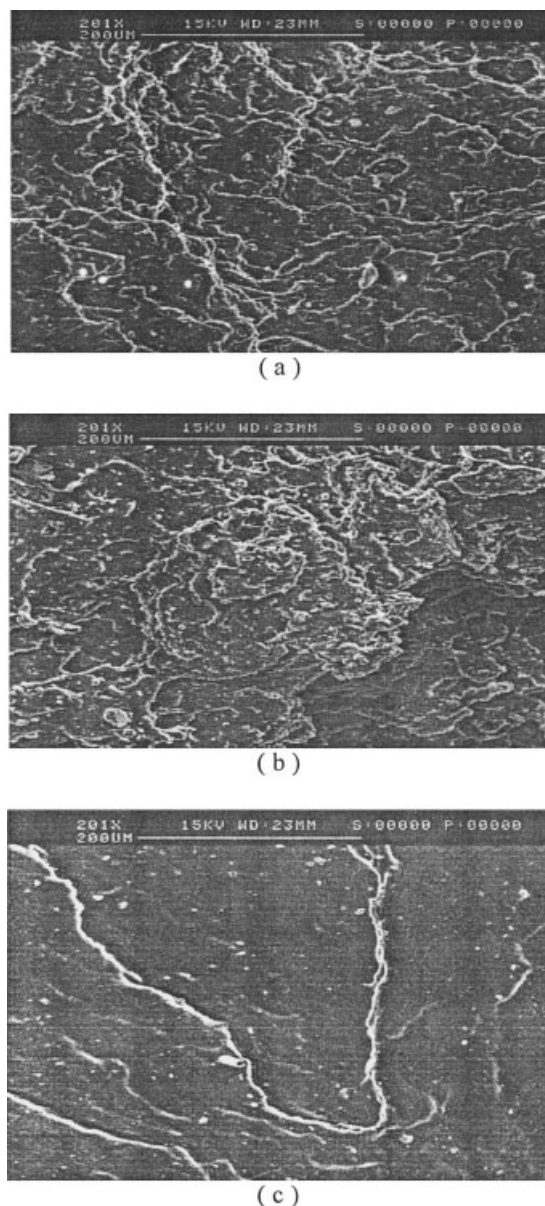
b



c

**Figure 13** SEM micrographs of tensile fracture surfaces of PVCv/NBR blends: (a) 80/20; (b) 50/50; (c) 20/80.





**Figure 14** SEM micrographs of tensile fracture surfaces of PVCr/NBR + AAc blends: (a) 80/20; (b) 50/50; (c) 20/80.

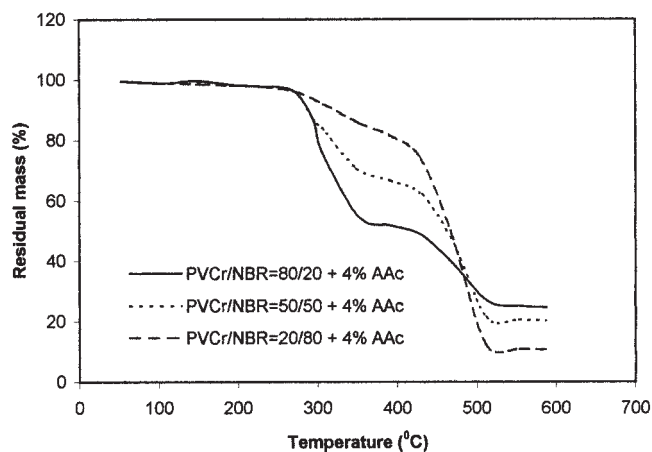
### Mechanical properties of blends

Figures 4 and 5 show the effect of blend composition on stress at break and stress at 100% elongation ( $M_{100}$ ) of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends. It can be seen that, as the NBR composition increases, both properties decrease as a result of decreasing blend rigidity. Increasing NBR composition in the PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends also increases the elongation at break ( $E_b$ ) (Fig. 6). As the NBR content increases, the stiffness and brittleness of the blends gradually decrease, with an associated increase in  $E_b$ . However at a similar blend composition, PVCr/NBR + AAc blends show higher stress at break and stress at 100% elongation ( $M_{100}$ ) but

lower elongation at break  $E_b$ , than those of PVCv/NBR blends. The better stress at break and stress at 100% elongation of PVCr/NBR + AAc over those of PVCr/NBR and PVCv/NBR blends is explained by the enhancement in interfacial adhesion between PVCr and NBR phases.

### Analysis of infrared spectroscopy

Figures 7 and 8 show the comparison of FTIR spectra of PVCr/NBR + AAc and PVCr/NBR blends at 80/20 (wt/wt). Both figures show the olefinic C—H stretching frequency around 2900–3000  $\text{cm}^{-1}$ . With respect to the  $\text{CH}_2$  bending vibration above 1435  $\text{cm}^{-1}$ , the peak appearing at around 2237  $\text{cm}^{-1}$  is attributed to the CN group from the NBR structure and the C—H out-of-plane bending frequency at 970  $\text{cm}^{-1}$ . In both figures the peak appearing around 1726 and 1723  $\text{cm}^{-1}$  was attributed to the free carbonyl group (C=O). However, the characteristic band of the PVCr/NBR + AAc at 1704  $\text{cm}^{-1}$  is assigned to the carbonyl groups (C=O). The correlation of both bands (free carbonyl group at around 1723  $\text{cm}^{-1}$  and carbonyl group at 1704  $\text{cm}^{-1}$ ) is evidence of hydrogen bonding.<sup>20</sup> The presence of hydrogen bonding in the PVCr/NBR + AAc blends, which improved the mechanical properties, might be a result of the grafting of AAc with PVCr in the blends. Figure 9 shows the reaction of AAc-grafted recycled PVC with NBR in the blends that occurred during melt mixing. The grafting of AAc with PVCr and NBR acts as an emulsifier at the interface and thus reduces the interfacial tension, leading to small and uniform distribution of the NBR phase. Snoppy et al.<sup>11</sup> also showed that the addition of compatibilizer to the polyolefin/nitrile rubber system not only reduces the dimension of the minor phase, but also results in uniform distribution of the minor phase.



**Figure 15** TG thermograms of PVCr/NBR + AAc blends.

**TABLE III**  
Data of Total Weight Loss and Weight Loss of PVCv/  
NBR Blends Obtained Using Thermal Gravimetry

Blend ratio	Total weight loss (%)	Weight loss at		
		300°C (%)	400°C (%)	500°C (%)
80/20	75.70	21.73	51.92	72.13
50/50	82.92	17.03	37.15	80.02
20/80	88.01	7.06	9.13	83.99

### Swelling behavior analysis

Figures 10 and 11 show the effect of blend composition on the swelling index of PVCr/NBR, PVCr/NBR + AAc, and PVCv/NBR blends in IRM 903 oil for 70 h and in toluene for 46 h. It can be seen in both figures that the swelling index increases with increasing NBR content in all blends, which is a result of the blend becoming less stiff and more penetrable by the IRM 903 oil and toluene. However, at a similar blend composition, the swelling index of PVCr/NBR + AAc blends is lower than that of PVCr/NBR and PVCv/NBR blends. Again, this result was attributed to the AAc grafting with PVCr and the enhancement in interfacial adhesion between PVCr and NBR phases.

### Morphology properties of blends

Figures 12(a)–(c), 13(a)–(c), and 14(a)–(c) show comparisons of tensile fracture surfaces of PVCr/NBR, PVCv/NBR, and PVCr/NBR + AAc blends at 80/20, 50/50, and 20/80 (wt/wt). It can be seen in all figures that, as the NBR content increases, the nature of failure surface changes from rough to smooth. However, at similar blend compositions, the PVCr/NBR + AAc blends [Figs. 14(a)–(c)] exhibit better strength with many tear lines and more homogeneous blends than that of PVCr/NBR [Figs. 12(a)–(c)] and PVCv/NBR blends [Fig. 13(a)–(c)]. Figure 12(a) shows a coarse and unstable particle structure and an uneven distribution of the dispersed NBR phase, which indicates low adhesion between phases, giving rise to poor stress transfer across the interface.<sup>21</sup> However, with the presence of AAc in PVCr/NBR blends [Fig. 14(a)], one can

observe better dispersion and more even distribution in the PVC matrix, indicating improvement in compatibility between PVCr and NBR. The presence of many tear lines on the tensile fracture surfaces of PVCr/NBR + AAc blends [Fig. 14(a)–(c)] indicates the effect of increased interaction between phases, thus improving compatibility and a consequent increase in the mechanical properties.

### Thermal degradation of the blends

Thermogravimetric analysis enables us to study the various loss of mass caused by an increase in temperature of the samples. Degradation occurs and the mass remaining decreases as the temperature increases. Figure 15 shows the typical thermogravimetric degradation of PVCr/NBR + AAc blends in the temperature range of 50–600°C. For all blend compositions, the degradation of PVCv/NBR, PVCr/NBR, and PVCr/NBR + AAc blends, which start at 220°C, was completed at 590°C. Tables III and IV show the total weight loss and weight loss at different temperatures of PVCv/NBR, PVCr/NBR, and PVCr/NBR + AAc blends. It can be seen that the total weight loss sequence of these blends can be given as PVCr/NBR < PVCr/NBR + AAc < PVCv/NBR blends, which indicates that the presence of AAc has reduced the thermal stability in the PVCr/NBR blends. A similar trend was observed by Qiao et al.,<sup>22</sup> who reported that the compatibilizer has a decreasing influence of thermal stability on the composite materials, especially stearic acid.

## CONCLUSIONS

The presence of AAc significantly improved the mechanical properties and swelling resistance of PVCr/NBR blends, as shown by stabilization torque, mixing energy, stress at break, stress at 100% elongation ( $M_{100}$ ), and lower elongation at break  $E_b$  and swelling index than those of PVCr/NBR and PVCv/NBR blends. The improvement in the interfacial adhesion between PVCr and NBR phases with the addition of AAc is clearly demonstrated in the morphology study

**TABLE IV**  
Data of Total Weight Loss and Weight Loss of PVCr/NBR and PVCr/NBR  
+ AAc Blends Obtained Using Thermal Gravimetry

Blend ratio	Total weight loss (%)		Weight loss at					
			300°C (%)		400°C (%)		500°C (%)	
	Without AAc	With AAc	Without AAc	With AAc	Without AAc	With AAc	Without AAc	With AAc
80/20	71.95	75.34	20.94	18.57	46.67	48.64	67.11	70.84
50/50	78.57	79.79	11.19	13.04	32.01	34.10	74.46	76.87
20/80	85.8	88.23	4.59	6.68	16.55	19.73	81.04	84.62

of tensile fracture surfaces of the blends. However, the presence of AAC reduced the thermal stability of PVCr/NBR blends.

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