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Continuous ultrasonic process for in situ compatibilization of polypropylene/natural rubber blends

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Abstract

The immiscible polypropylene (PP)/natural rubber (NR) blends of various concentrations were prepared by using a twin-screw extruder. The prepared blends were passed through the reactor where they were ultrasonically treated by an extrusion process. Mechanical properties and rheology of the obtained blends were studied, along with morphology by using the scanning electron microscopy and the atomic force microscopy (AFM). Mechanical properties of the treated blends were found to improve significantly in comparison with those of untreated blends. Under most treatment conditions, no significant differences in the viscosity of the treated and untreated blends were observed. The AFM studies revealed the development of interfacial layers, interfacial roughening and improved interfacial adhesion between PP and NR phases in the blends subjected to ultrasonic treatment. At the same time weak adhesion and delamination at the interface were found in the untreated blends. The improved interfacial adhesion, morphology and mechanical properties are believed to be due to the formation of in situ copolymer at the interface of two immiscible polymers caused by an ultrasonic treatment without the use of any chemicals.

Keywords: Compatibilization; Polypropylene/natural rubber blends; Ultrasound

1. Introduction

Polymer blending is an effective way to achieve a desirable combination of properties that are often absent in single component polymers. However, simply mixing two or more polymers together will not ensure the blend with desirable properties because of the incompatibility. Compatibilization of polymer blends has been studied for decades [1]. There are two general routes to improve compatibility. One is adding a third component, typically a copolymer, which is miscible with each homopolymer. The other is inducing a chemical reaction, leading to a modification of the polymer interface in two-phase blends. Reactive blending is utilized to improve the compatibility of the blends. The in situ formed compatibilizers have segments that are chemically identical to their respective counterparts in the unreacted homopolymers and are thought to be located preferentially at the interface. Many researchers have studied in situ compatibilization [2–6]. Lyu et al. [7] and Lee et al. [8] studied interfacial roughening in polymer blends induced by the formation of copolymer.

Thermoplastic elastomers play a more and more important role in polymer industry due to their good processability and their elastomeric properties [9]. Among them, polypropylene (PP)/natural rubber (NR) blends have been studied due to the high melting temperature of PP meeting the dimensional stability at elevated temperatures and the low cost and abundant supply of NR [10–15].

The application of ultrasound to polymer systems is an attractive field of study. In particular, copolymerization by prolonged ultrasound irradiation in solutions has been studied [16–22]. Recently, high power ultrasound waves have been discovered to compatibilize polymer blends in the melt state during continuous extrusion at very short residence time under high pressure and temperature [23,24].

In the present study, in situ compatibilization of immiscible PP/NR blends of various concentrations was investigated by means of an extrusion process using a new ultrasonic reactor. Mechanical properties and rheology of the obtained blends were studied, along with morphology by

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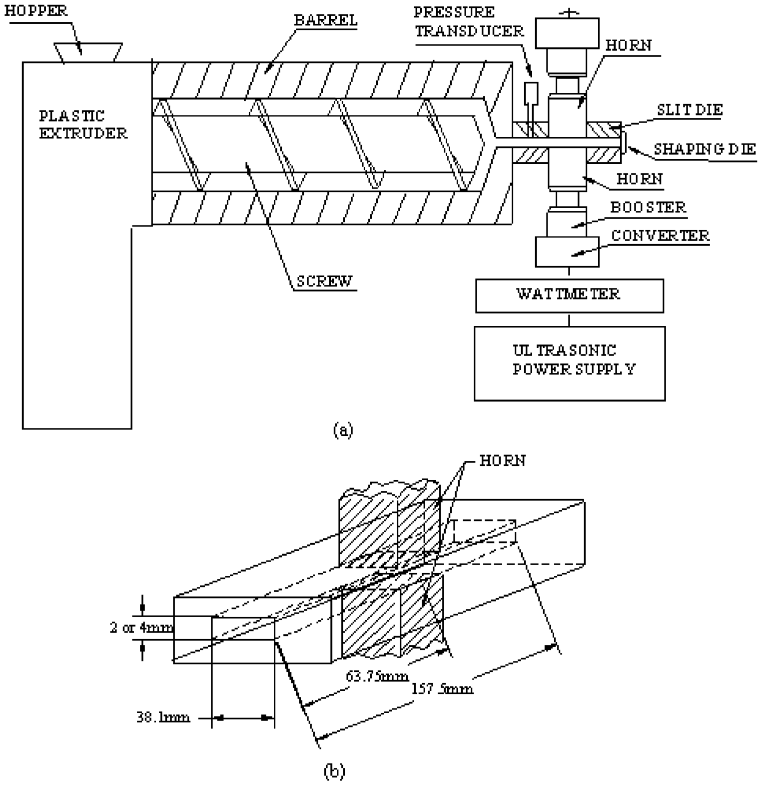


Fig. 1. Schematic drawing of the ultrasonic reactor with a slit and shaping die (a) and the slit die dimensions with positions of horns (b).

using scanning electron microscopy (SEM) and atomic force microscopy (AFM). The development of interfacial roughening and improved interfacial adhesion between PP and NR phases in the blends subjected to ultrasonic treatment was reported.

2. Experimental

2.1. Materials

The NR used in the experiments was SMR CV60 obtained from Akrochem Corp. ($M_w = 1,116,000$, polydispersity index = 6.19). The PP used in this study was Pro-fax 6523 from Montell, Inc. ($M_w = 351,000$, polydispersity index = 4.3, melt flow index = 4.1 dg/min).

2.2. Preparation of blends

A co-rotating twin screw extruder (JSW Labotex30) was used to prepare PP/NR blends of 25/75, 50/50, and 75/25 wt.% composition. PP and NR were physically mixed and then fed to the extruder. Screw speed was set at 180 rpm and zone temperatures of 165/165/175/180/180/185/190 °C were used. The extrudates of the prepared mixtures were cooled, pelletized and then dried in a vacuum oven for 24 h at a temperature of 60 °C.

2.3. Ultrasonic reactor and blends treatment

The ultrasonic reactor consists of a 25.4 mm plastic extruder (Killion) followed by a shaping die having four holes of diameter of 3.175 mm and length of 17 mm, which were attached to the end of the slit die with dimensions of $157.5 \times 38.1 \times 6\ \mathrm{mm}^3$. Two water-cooled horns of square cross sections of $38.1 \times 38.1\ \mathrm{mm}^2$ were placed in the middle of the slit die. A schematic drawing of the reactor and the slit die dimensions are shown in Fig. 1. A pair of 3.3 kW ultrasonic power supplies, converters, and 1:1 boosters provided the longitudinal vibrations of the horn with a frequency of 20 kHz and various amplitudes. A pressure transducer (Dynisco) of maximum pressure of 51.7 MPa was placed in the slit die before the treatment zone at a distance of 21.6 mm from the front of the horn. One or both horns can be moved vertically inside of the slit die to create controllable gap between horn faces in which the material was subjected to compressive ultrasonic waves.

The PP/NR blends were then fed into the ultrasonic reactor at flow rates of 0.32 g/s and 0.63 g/s. The gaps between the two horns were 2 and 4 mm. The mean residence times in the treatment zone corresponding to these gaps were, respectively, 4.1 s and 8.2 s at a flow rate of 0.63 g/s. The screw speed was 100 rpm. The barrel temperature was set at 178 °C. The amplitudes used were 5, 7.5, and 10 micrometers. One horn or two horns were used. The treated blends were cooled by water and then dried in a vacuum oven for 24 h at a temperature of 60 °C.

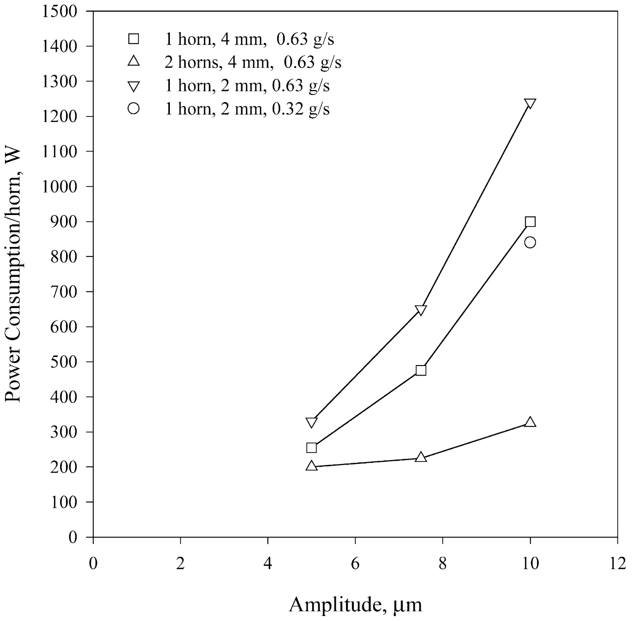


Fig. 2. Power consumption per horn as a function of amplitude for 50/50 PP/NR blends.

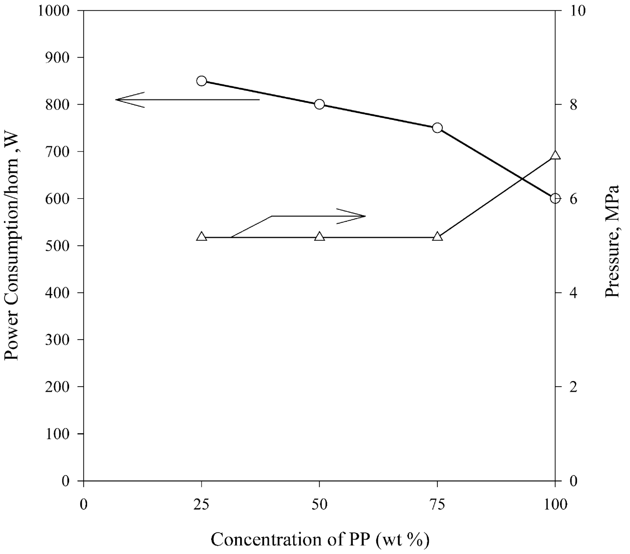


Fig. 4. Power consumption and pressure as a function of PP concentration during treatment of PP and PP/NR blends with1 horn at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.

The obtained blends were compression molded into slabs of dimensions of 127 × 127 × 2 mm^3 for 5 min at a temperature of 180 °C and at a pressure of 13.8 MPa in an electrically heated compression molding press (Wabash). The molded samples were cooled under compression to maintain dimensional stability and flatness.

2.4. Measurements of mechanical properties

An Instron tensile tester, Model 5567, was used for the stress–strain measurements on specimens cut from the molded slabs. All tests were performed at room temperature with a crosshead speed of 500 mm/min (ASTM D412).

Impact testing was performed on discs cut from the mold slabs with dimensions of a diameter of 74 mm and a thickness of 3 mm using a dart impact tester (Dynatup 8250) with a velocity of 4.17 m/s and a dart weight of 5.08 kg. The diameter of the hemispherical probe was 12.7 mm and samples were held in place on an annular ring with an internal diameter of 38.1 mm. All samples were totally penetrated. All tests were performed at room temperature.

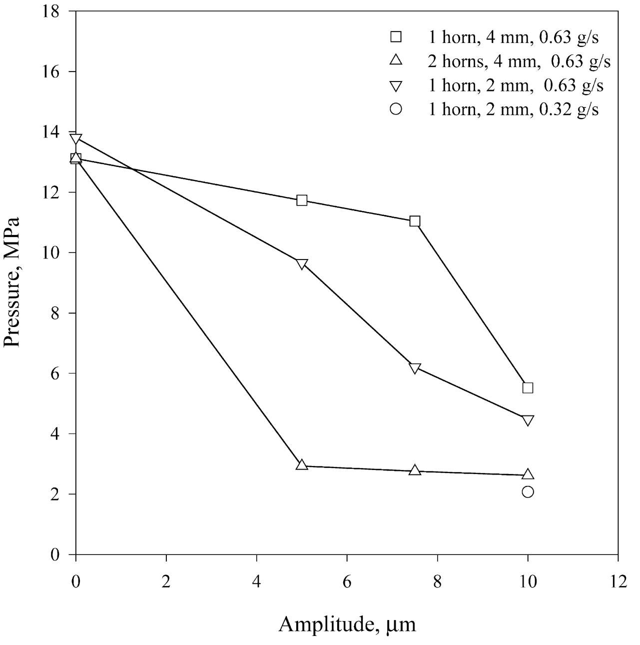


Fig. 3. Pressure as a function of amplitude for 50/50 PP/NR blends.

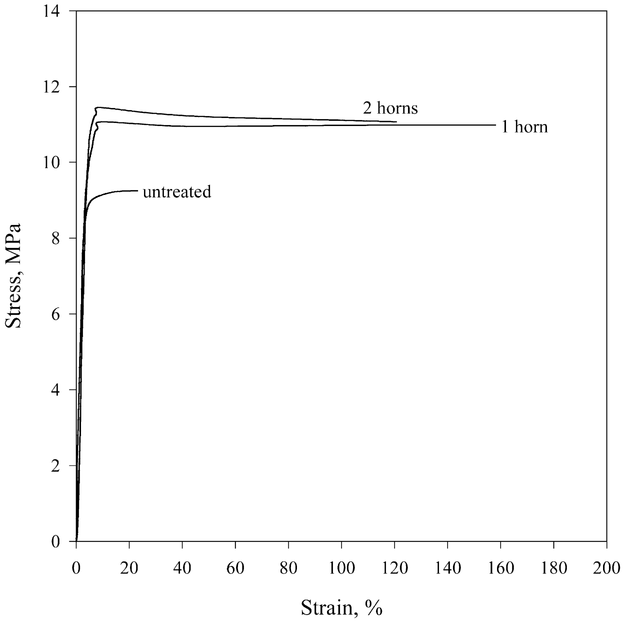


Fig. 5. The stress–strain curves for 50/50 PP/NR blends untreated and treated at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.

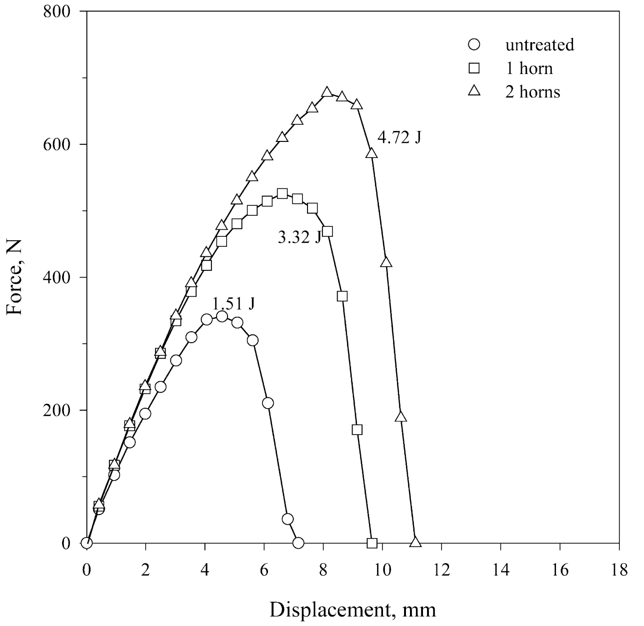


Fig. 6. Impact force vs. displacement and energy for probe penetration for 50/50 PP/NR blends untreated and treated at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.

2.5. Measurements of rheological properties

The rheological behaviors of the blends and NR were investigated using an Advanced Polymer Analyzer (APA 2000, Alpha Technologies) at a temperature of 200 °C and a strain amplitude of 7.1%. The biconical rotor with an angle of 7° and diameter of 63.5 mm was used. ARES (Rheometric Scientific) was used to measure the dynamic viscosity of PP using the parallel plate mode. The diameter of the disk was 25 mm.

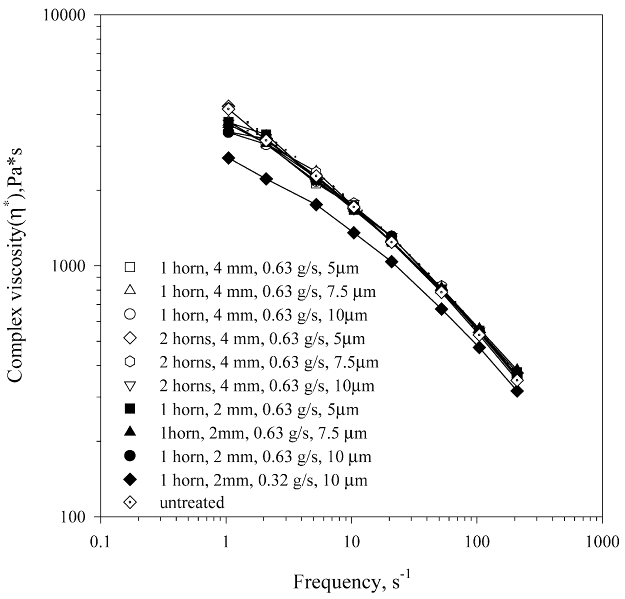


Fig. 7. Complex dynamic viscosity as a function of frequency for untreated and treated 50/50 PP/NR blends at 200 °C.

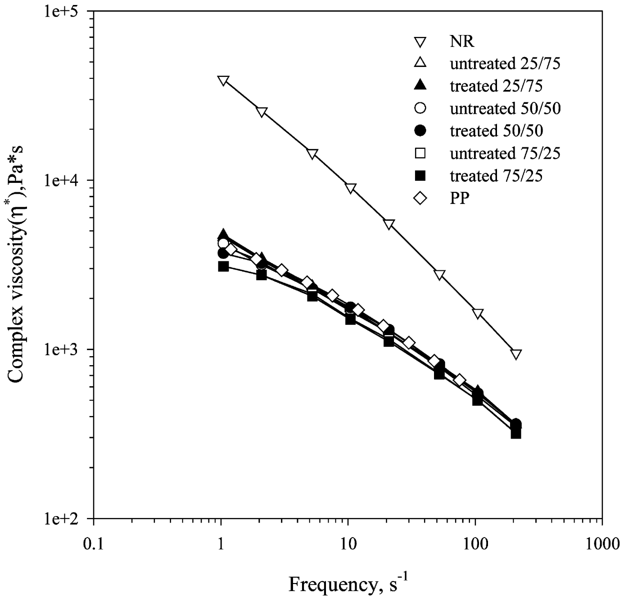


Fig. 8. Complex dynamic viscosity as a function of frequency for NR, PP, and various compositions of PP/NR blends at 200 °C untreated and treated with one horn at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.

2.6. Morphological studies

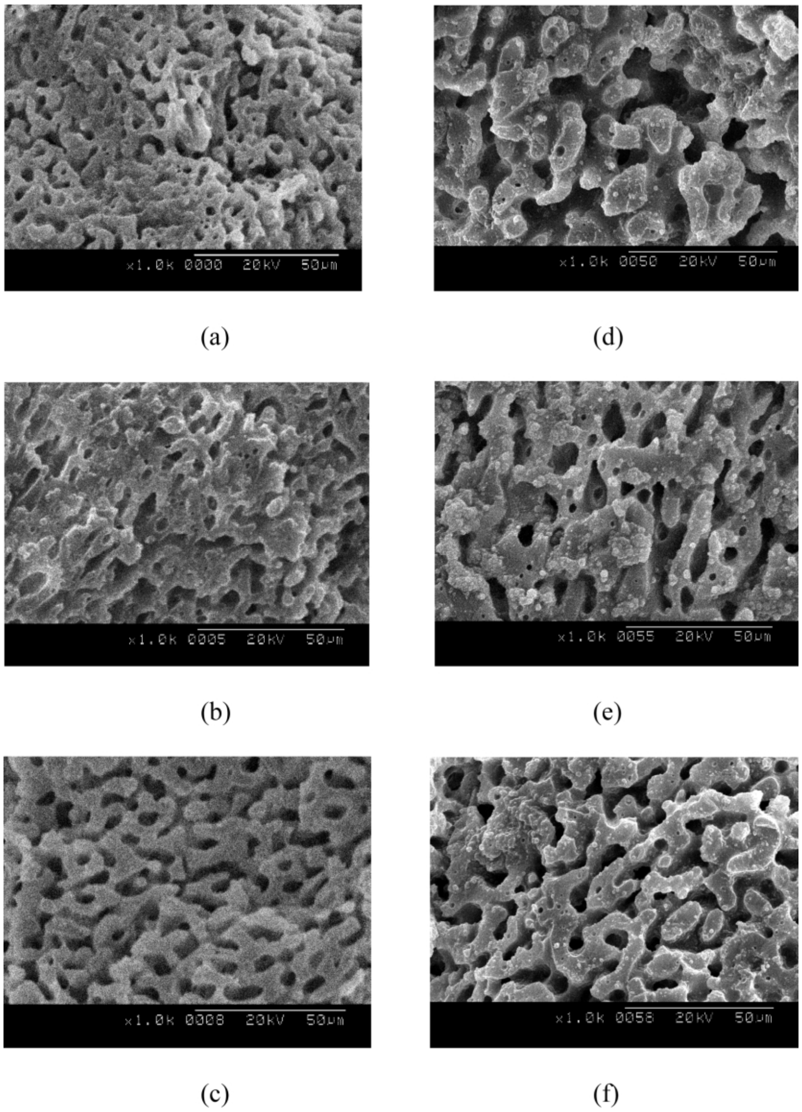
The phase morphologies of the blends were studied by SEM (model Hitachi S-2150) and AFM (Nanoscope IIIa, Digital Instrument, Inc.).

The SEM samples were prepared as follows: the prepared blends were fractured in liquid nitrogen and the NR phase was extracted in benzene at 50 °C for 12 h. After etching, the samples were dried in a vacuum oven at 70 °C for 12 h and were coated with silver using sputter coater. Also, the blends were annealed at a temperature of 190 °C for 10 min in compression molding press. The SEM samples of the annealed blends were prepared using the same procedure.

AFM was used to test the hypothesis of copolymer formation at the interface due to the ultrasonic treatment. To study the morphology of the bulk, the cross-sections were cryo-microtomed perpendicular to the surface of the plaques and the observed directly. The AFM images were obtained in air. The tapping mode was used. Measurements were performed at ambient conditions using rectangular type Si probes with a spring constant of $50\,\mathrm{N\,m}^{-1}$ and resonance frequencies in the 284–362 kHz range. The tip radius was 10 nm. The AFM topographic (height) and the elastic (phase) images were simultaneously obtained under normal and hard tapping conditions on the microtomed surface of PP/NR blends. Phase images revealed hard regions in bright (the PP phase) and soft regions in dark (the NR phase).

Table 1
The tensile properties of PP/NR blends

Composition of PP/NR	Treatment condition	Tensile strength (MPa)	Elongation at break (%)	Young's modulus (MPa)	Toughness (MPa)
25/75	Untreated	4.93	63.7	70.0	2.68
	1 horn, 4 mm, 0.63 g/s, 10 μm	4.17	50.9	51.8	1.99
50/50	Untreated	9.20	23.0	233	1.98
	1 horn, 4 mm, 0.63 g/s, 5 μm	9.26	46.5	245	4.04
	1 horn, 4 mm, 0.63 g/s, 7.5 μm	10.1	76.1	243	8.36
	1 horn, 4 mm, 0.63 g/s, 10 μm	10.3	161	243	16.43
	2 horns, 4 mm, 0.63 g/s, 5 μm	10.4	99.0	228	10.05
	2 horns, 4 mm, 0.63 g/s, 7.5 μm	9.85	49.9	271	4.61
	2 horns, 4 mm, 0.63 g/s, 10 μm	11.2	130	296	14.48
	1 horn, 2 mm, 0.63 g/s, 5 μm	11.1	38.2	328	4.17
	1 horn, 2 mm, 0.63 g/s, 7.5 μm	10.3	97.8	300	10.32
	1 horn, 2 mm, 0.63 g/s, 10 μm	11.2	31.9	294	3.43
	1 horn, 2 mm, 0.32 g/s, 10 μm	6.40	3.49	206	0.13
75/25	Untreated	11.9	11.6	305	1.29
	1 horn, 4 mm, 0.63 g/s, 10 μm	16.8	26.2	483	4.24

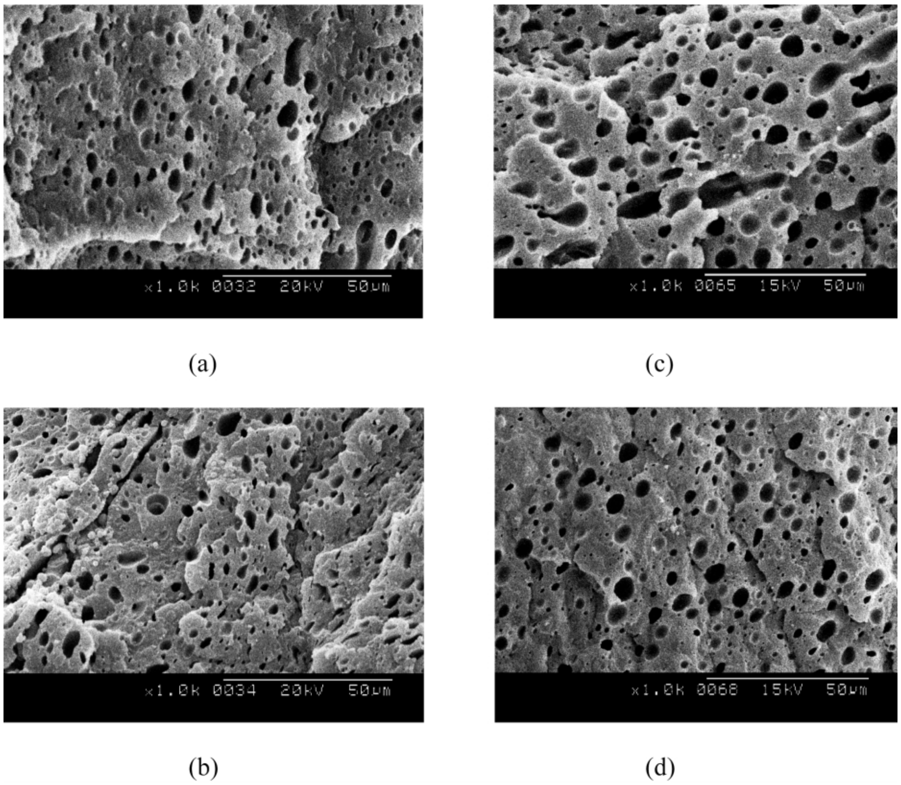


(a) (d)

(b) (e)

(c) (f)

Fig. 9. The SEM photomicrographs of 50/50 PP/NR blends before (a), (b), (c) and after (d), (e), (f) annealing for untreated (a), (d) and treated with one horn (b), (e) and two horns (c), (f) at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.



(a) (c)

(b) (d)

Fig. 10. The SEM photomicrographs of 75/25 PP/NR blends before (a), (b) and after (c), (d) annealing for untreated (a), (c) and treated with one horn (b), (d) at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 µm.

3. Results and discussions

3.1. Power consumption and die pressure

Fig. 2 shows the power consumption vs. ultrasonic amplitude at various gaps, and flow rates. Using one or two horns, the power consumed during treatment increases with the increase of amplitude, flow rate (one data point is indicated for 0.32 g/s) and decrease of gap. The power consumption per horn in the case of two horns is much lower than that in the case of one horn evidently due to an interaction of ultrasonic waves. In order to apply ultrasonic treatment on an industrial scale, one of the most important problems is the scale-up of the process such that the process would become economically viable. However, there is a limit in the productivity of the process due to a limitation in the delivery of high power output by available ultrasonic generators. Therefore, the use of two or more horns could be an important step for scale-up of the process as discussed in the patent dealing with ultrasonic devulcanization [25].

Fig. 3 shows dependencies of the die pressure on ultrasonic amplitude at various gaps and flow rates using one or two horns. It was observed that the die pressure decreases upon the application of ultrasound and the pressure decreases further with increasing amplitude. The use of two horns reduces the pressure significantly in comparison with the use of one horn, indicating that the treatment is facilitated. This decrease of pressure after imposition of ultrasound has been previously explained as the combined effect of the softening of the materials and a possibility of slippage of the melt along the die wall due to ultrasonic vibrations [26]. The pressure decreases with the decrease of flow rate and gap. The observed effect of gap during treatment is quite unusual. Typically, at a lower gap without imposition of ultrasound the pressure is higher than that of a larger gap. Also, at the same flow rate the mean residence time is longer at a larger gap. Therefore, due to the longer residence time the effect of ultrasound in a larger gap should be expected to be greater. However, for a larger gap at the same amplitude, the strain amplitude mainly determining the effect of ultrasound on polymers is lower. Evidently, due to the increase of the strain amplitude, the effect of ultrasound amplitude on reduction of viscosity of melt is much stronger at a smaller gap than at a larger gap, even though the residence time is shorter in this smaller gap. This led to the observed pressure decrease at a lower gap.

Fig. 4 shows dependency of the power consumption and the die pressure on PP concentration for the ultrasonically treated blends with one horn at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 µm. The power consumption decreases with the increase of PP concentration, while die pressure is independent of PP concentration in the blends and only increases for pure PP.

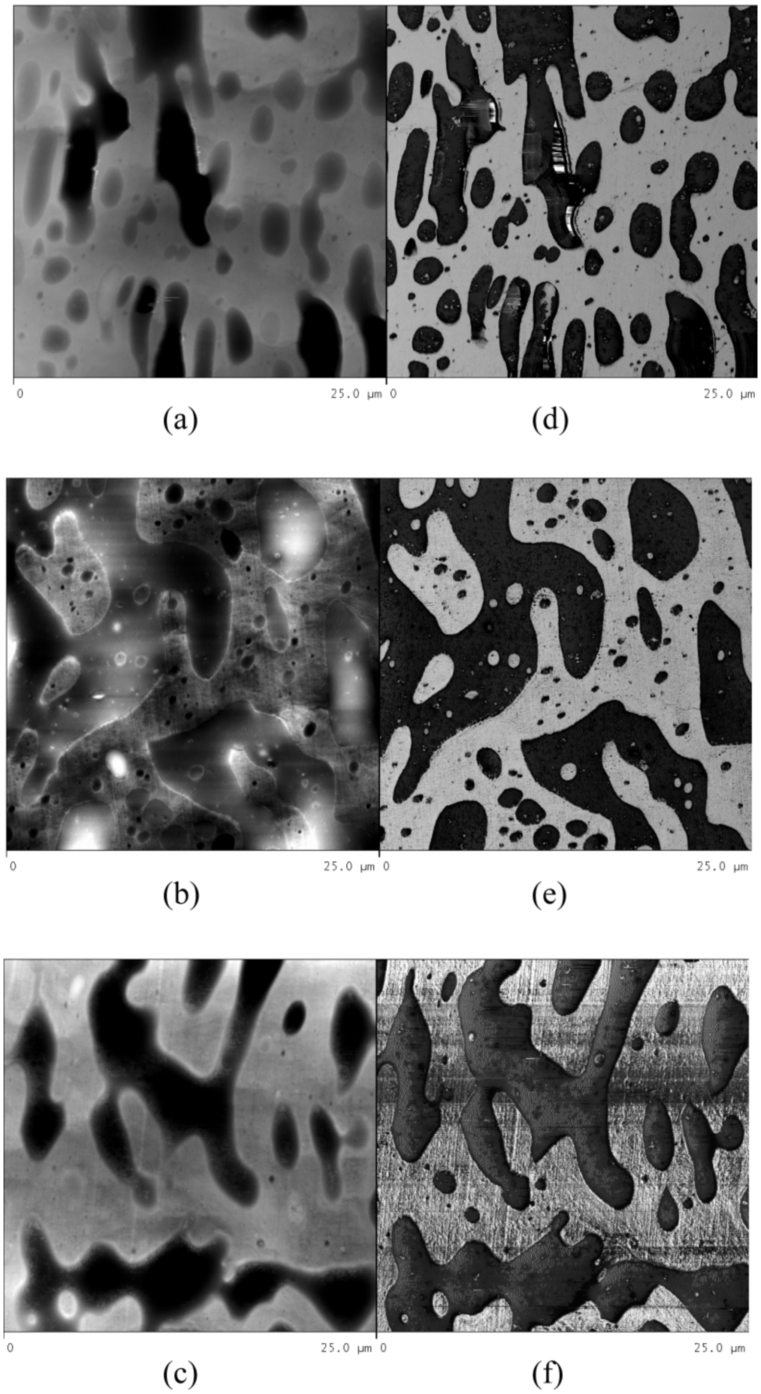
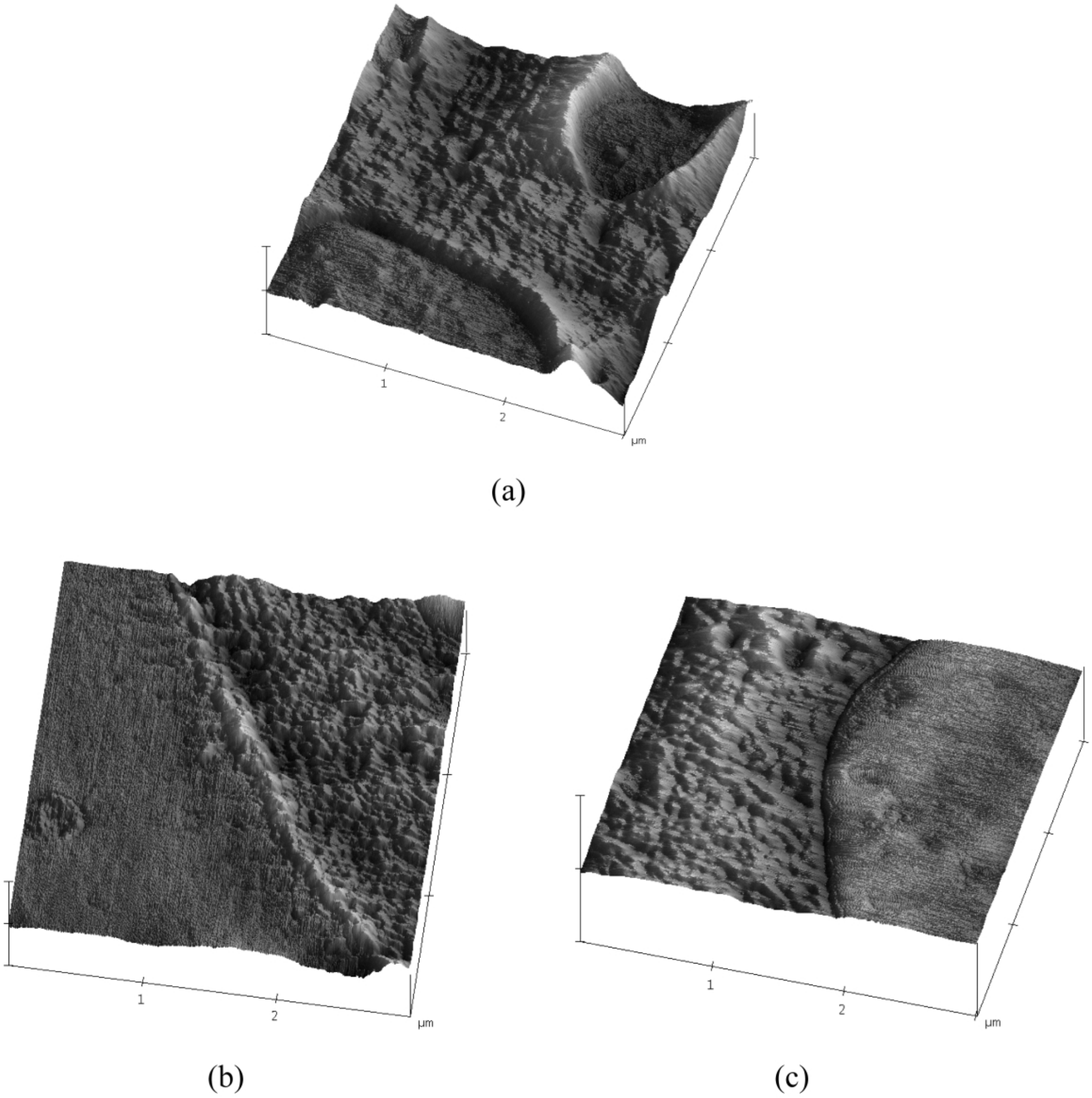


Fig. 11. The AFM height (a), (b), (c) and phase (d), (e), (f) images of 50/50 PP/NR blends for untreated (a), (d) and treated with one horn (b), (e) and two horns (c), (f) at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 µm.

3.2. Mechanical properties

Fig. 5 shows the stress–strain behaviors for 50/50 PP/NR blends untreated and treated at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 µm. Table 1 shows the tensile strength, Young's modulus (secant modulus at 3% elongation), the elongation at break, and the toughness of PP/NR blends of various concentrations obtained at various conditions of treatment. It is seen that under most of conditions, ultrasonic treatment of the blends during extrusion significantly increased these properties of each blend as compared to the untreated blend. The primary products of the sonochemical cleavage of polymers are macromolecular radicals [27]. The radicals can combine to form a copolymer. It is believed that the ultrasonic treatment improves the compatibility and interfacial adhesion between the two phases due to the formation of in situ copolymer at the interface of two immiscible polymers



(a)

(b)

(c)

Fig. 12. The three-dimensional AFM surface profile of 50/50 PP/NR blends for untreated (a) and treated with one horn (b) and two horns (c) at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.

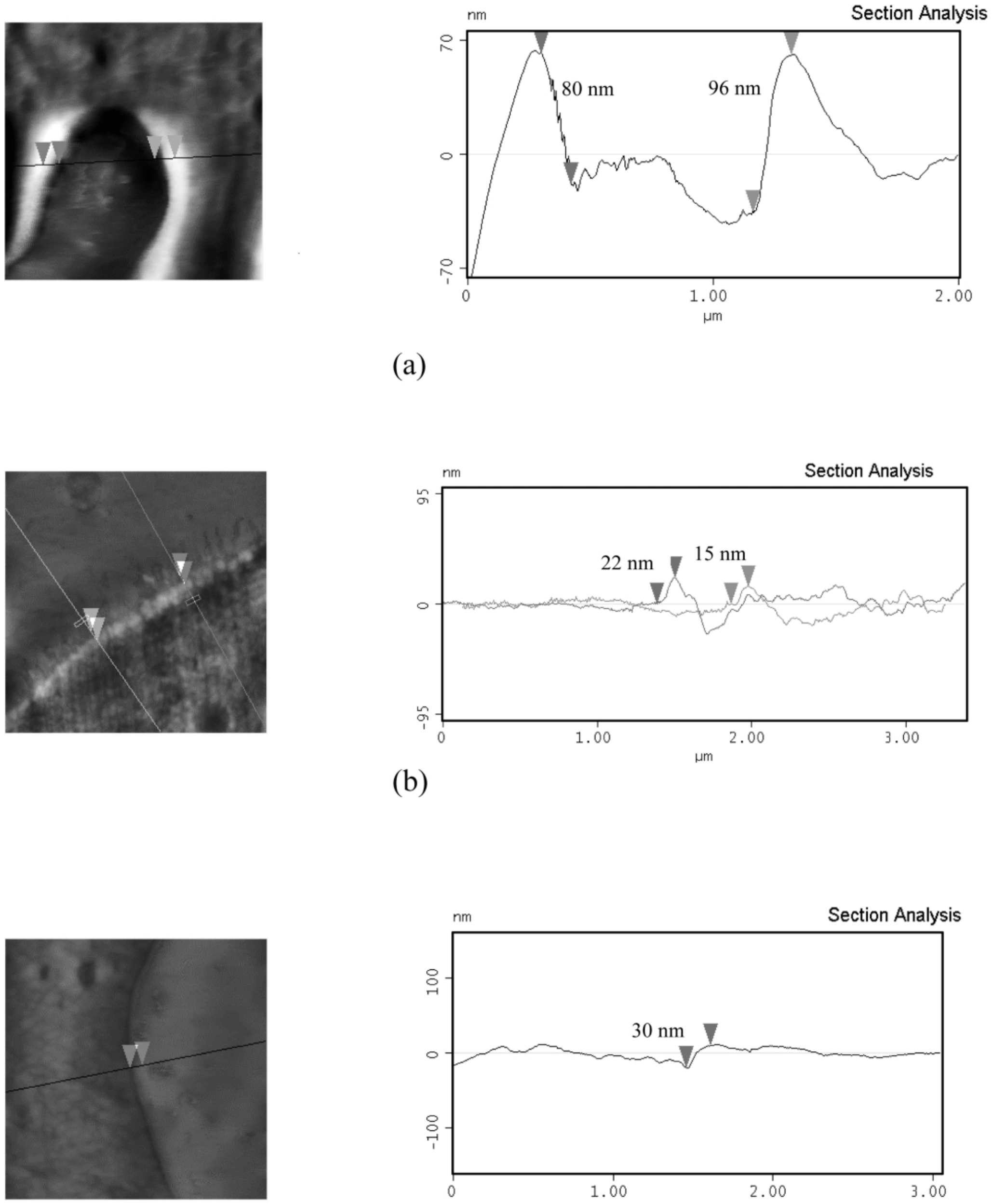
without the use of any chemicals. This will be discussed further in the morphology section. As seen in the cases of 25/75 PP/NR and longer mean residence time in the ultrasonic treatment zone (a flow rate of 0.32 g/s) of 50/50 PP/NR, the properties are worse than those of untreated blends due to the possible degradation of the main chains caused by the over-treatment.

Fig. 6 shows the impact behavior of untreated and treated PP/NR (50/50) blends at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm. This figure indicates the impact force felt at the probe of the falling dart plotted against its displacement through the sample. The values of the impact energy determined by the area under the force-displacement curve are given next to the curves. It is also believed that impact properties are significantly improved due to the formation of in situ copolymer at the interface.

3.3. Rheology

Fig. 7 shows the dependency of the complex dynamic viscosity on frequency for 50/50 PP/NR blends obtained at various treatment conditions at 200 °C. It was observed that there were no significant differences in the viscosity between untreated and most of treated blends at a wide range of shear rates and only the viscosity at the lowest frequency is affected by the treatment condition. Only at a flow rate of 0.32 g/s the viscosity is significantly decreased in the whole range of shear rates. This is due to the fact that a longer mean residence time in the ultrasonic treatment zone during extrusion caused a significant breakup of the main chains.

Fig. 8 shows the dependency of the complex dynamic viscosity on frequency for NR, PP, and various compositions of PP/NR blends at 200 °C untreated and treated



(a)

(b)

(c)

Fig. 13. The section analysis of 50/50 PP/NR blends for untreated (a) and treated with one horn (b) and two horns (c) at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.

under the treatment condition of one horn, a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm. Surprisingly, the viscosity of 25/75 PP/NR is only slightly higher than that of 50/50 PP/NR at low frequency region. It is believed that NR degraded a lot during compounding in the twin screw extruder. The viscosity behavior of the treated blends is similar to that of the untreated blends for 75/25 and 25/75 PP/NR blends in the whole range of frequencies and differs for 50/50 PP/NR blend at high frequency range only. NR, which was not extruded, show much higher viscosity than that of the blends. This confirms the degradation of the untreated blends during compounding which took place mainly due to the degradation of NR. The viscosity of untreated PP is similar to that of 50/50 PP/NR blend.

3.4. Morphology

Fig. 9 shows the SEM photomicrographs before and after annealing of 50/50 PP/NR blends untreated and treated at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm. There is no major change of morphology in

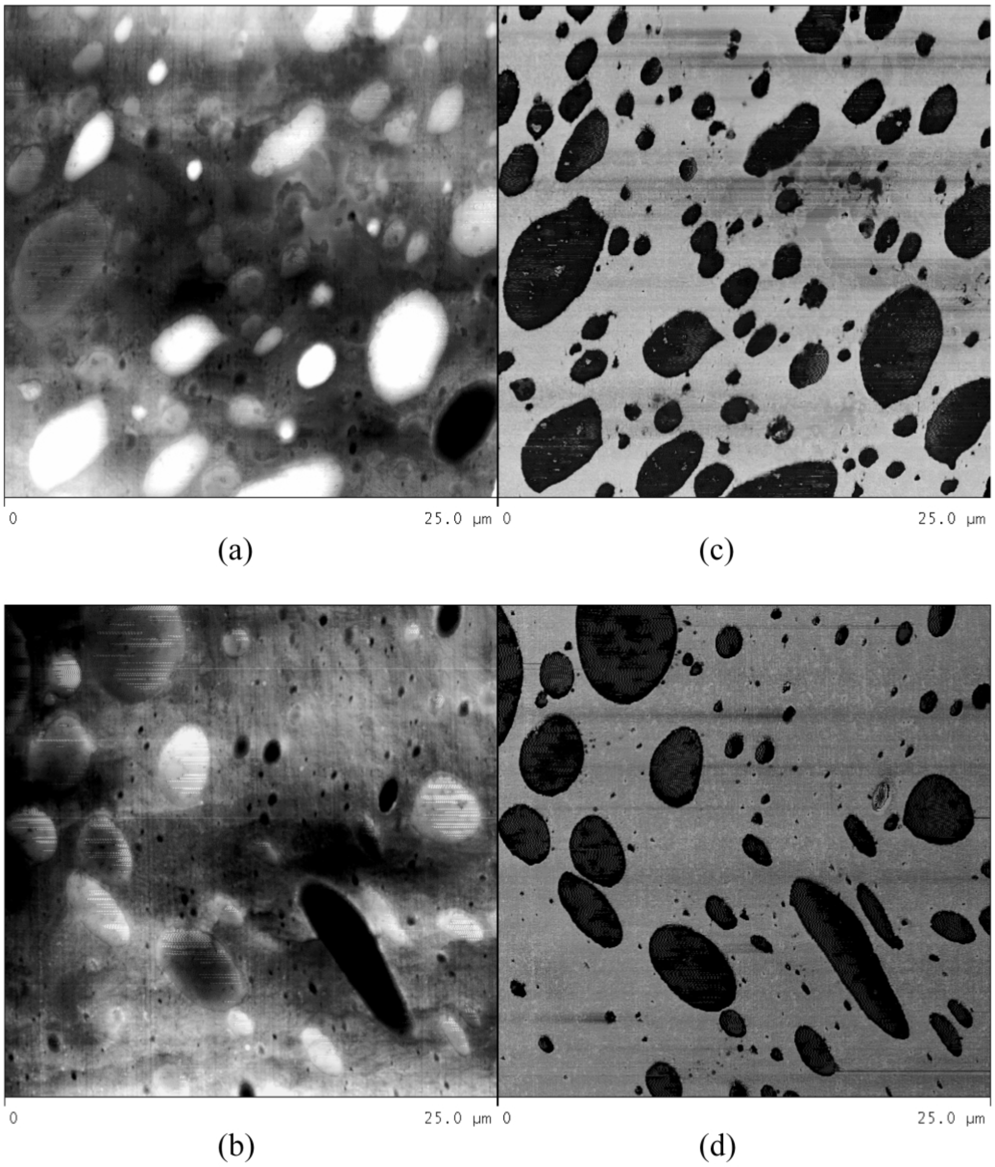
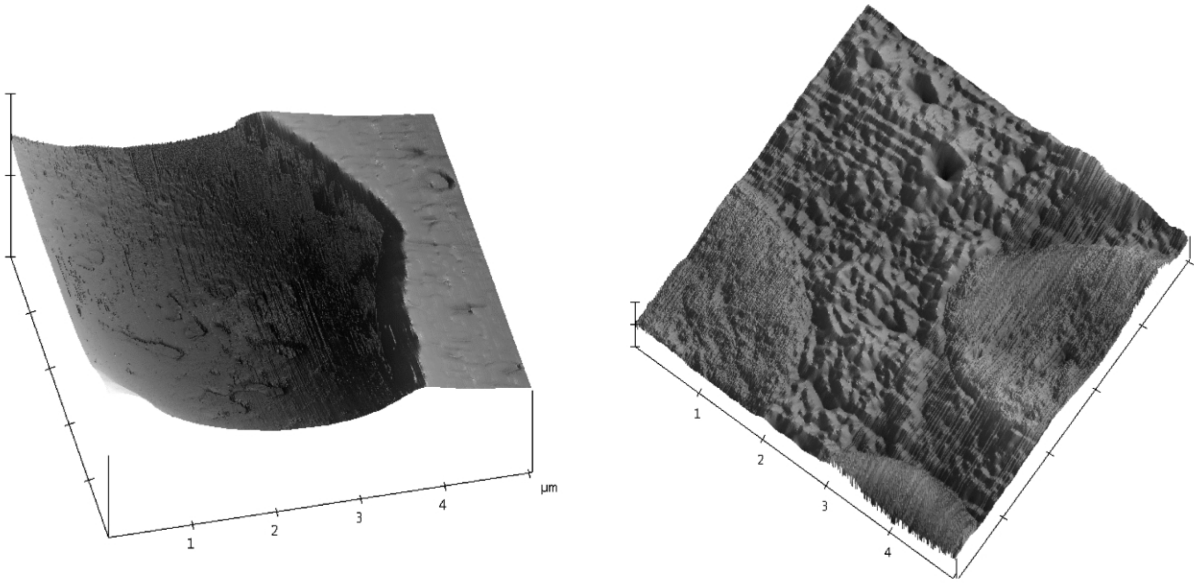


Fig. 14. The AFM height (a), (b) and phase (c), (d) images of 75/25 PP/NR blends untreated (a), (c) and treated with one horn (b), (d) at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.

untreated and treated blends before annealing. However, after annealing the domain size of the NR dispersed phase in treated blends is much smaller than that in untreated blends. It is believed that the retardation of phase growth in the treated blend takes place and the morphology of the treated blends is more stable than that of the untreated blend due to the copolymer formed during ultrasonic treatment. The copolymer leads to enhanced chemical interaction at the interface and improved adhesion between the two polymers. This in situ copolymer formation explains the superior mechanical properties of treated blends in comparison with those of untreated blends.

Fig. 10 shows the SEM photomicrographs before and after annealing of 75/25 PP/NR blends untreated and treated at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm. The change of morphology upon annealing of 75/25 PP/NR blends is clearly similar to that of 50/50 PP/NR blends. Again, the treated blends show stable morphology after annealing.

AFM was used to evaluate the effect of ultrasonic treatment on the interface morphology. Fig. 11 shows, respectively, the AFM height and phase images of 50/50 PP/NR blends untreated (a), (d), treated with one horn (b), (e), and treated with two horns (c), (f) at an amplitude of 10 μm, a gap of 4 mm, and a flow rate of 0.63 g/s. NR (dark phase) is seen to be dispersed in the bright PP matrix. In the untreated blend NR phase forms the ellipsoids and complex-shaped domains with a broad size distribution. The



(a) (b)

Fig. 15. The three-dimensional AFM surface profile of 75/25 PP/NR blends for untreated (a) and treated with one horn (b) at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.

boundaries between PP and NR phases in the untreated blend are very sharp. We can see the delamination of PP phase from the rubber phase at some places. In a contrast to the untreated blend, phase images of the blend treated with one horn do not reveal the delamination regions at the interface between the PP and NR phases. Phase images of the blend treated with two horns are similar to those of the blend treated with one horn.

Figs. 12 and 13 show, respectively, the three-dimensional AFM surface profile and the section analysis of the AFM images shown in Fig. 11. A three-dimensional AFM surface profile and a section analysis represent the interfacial topology between PP and NR phases. There are sharp steps developed at the interface between PP and NR in the untreated blend. The surface topology at each side of the step is relatively flat. These sharp steps as well as the presence of delamination regions indicate the poor adhesion between PP and NR phases. PP and NR have different thermal expansion coefficients and expand differently after the cryo-microtoming procedure. Because of the weak adhesion at the interface between PP and NR, sharp steps have been developed upon stress-relaxation. The height of the steps is about 100 nm. In a contrast to the untreated blend, a three-dimensional surface profile does not reveal sharp steps at the interface in the treated blends. The sectional analysis illustrates a smooth step with the height about 15–20 nm in the blend treated with one horn and 30 nm in the blend treated with two horns, which are almost an order of magnitude smaller compared to the untreated blend. Furthermore, AFM three-dimensional images reveal some interfacial roughening (interfacial curvature) at the interface between PP and NR in treated blends. This indicates much better adhesion at the interface in the PP/NR treated blends. The development of the interfacial roughening as well as improvement of an interfacial adhesion in an immiscible PET/PS blend due to the formation of copolymer upon addition of a reactive compatibilizer was reported by Lee et al. [8]. Lyu et al. [7] have also reported interfacial reaction between two immiscible polymers due to the creation of in situ copolymer. Thus, the AFM observation allowed us to visualize the interfacial topology changes due to the formation of in situ PP/NR copolymer by means of an ultrasonic treatment without use of any chemicals. The formation of the copolymer at the interface creates the interfacial roughness and eventually glues the two phases together, which prevents the step formation.

Fig. 14 shows the AFM height (a), (b) and phase (c), (d) images of 75/25 PP/NR of untreated (a), (c) and treated (b), (d) with one horn at an amplitude of 10 μm, a gap of 4 mm, and a flow rate of 0.63 g/s. Figs. 15 and 16 show, respectively, the three-dimensional AFM surface profile and the section analysis of the AFM images shown in Fig. 14. NR phase represents the dispersed phase and PP forms a matrix. As in the case of 50/50 PP/NR blends three-dimensional AFM images of 75/25 PP/NR blends exhibit sharp steps at the interface between PP and NR with a height

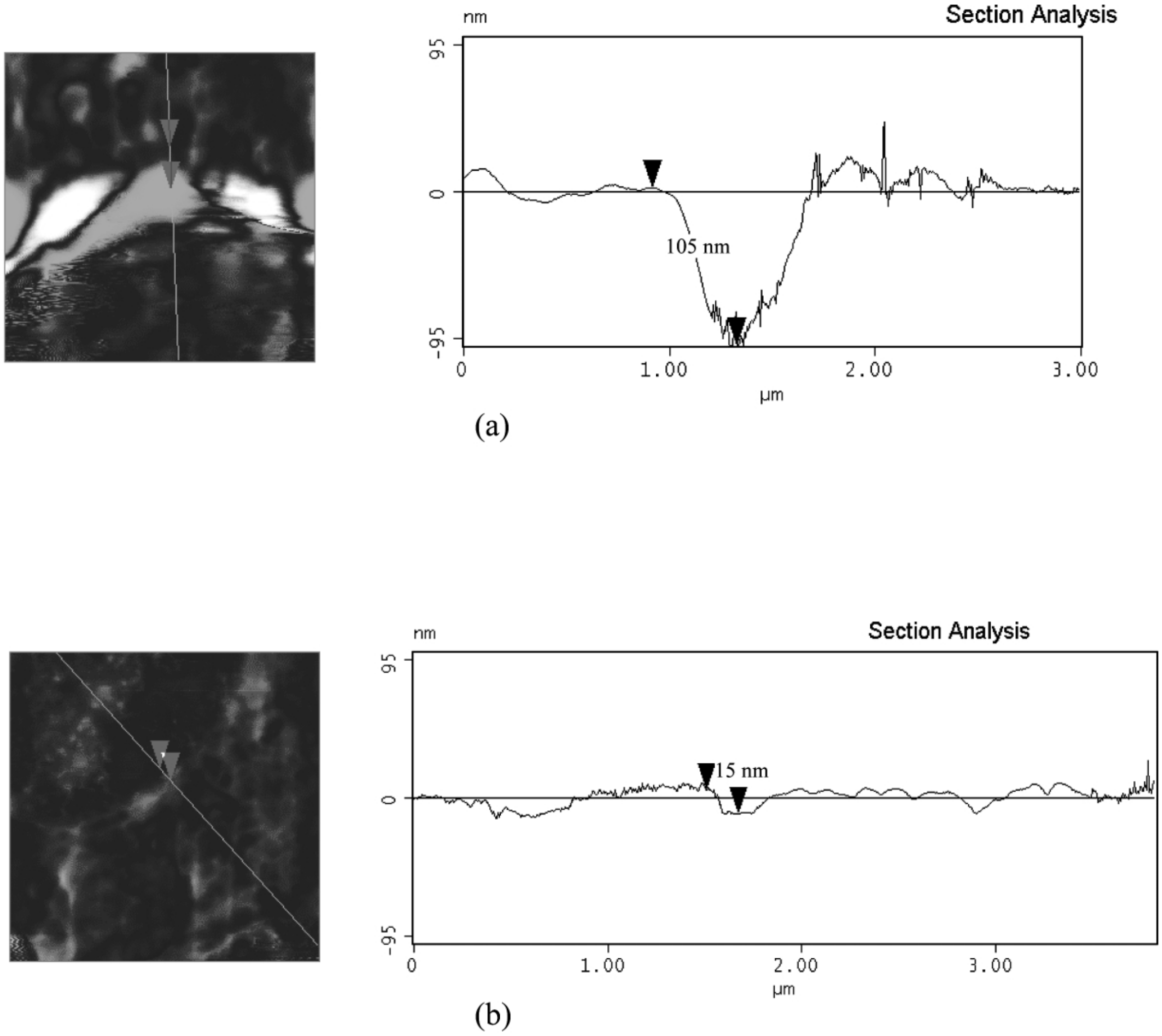


Fig. 16. The section analysis of 75/25 PP/NR blends for untreated (a) and treated with one horn (b) at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.

around 100 nm in the untreated blend and exhibit small (smooth) steps at the interface with the height about 15 nm. There is no delamination at the interface. This indicates good adhesion and formation of the copolymer.

It was interesting to find if there is a change of the thickness of the interfacial layer created by in situ formed copolymer. In this regard, Fig. 17 shows the height of step at the interface (a) along with the thickness of the interfacial layer (b) 50/50 PP/NR blends untreated and treated with one horn and two horns at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm. These data are obtained by averaging the results obtained on section profiles. It is seen that the error bar in the measurements is quite small in comparison with the measured values. These data clearly show that the height of step is significantly reduced while the thickness is appreciably increased after ultrasonic treatment indicating in situ formation of a PP/NR copolymer upon ultrasonic treatment of the blends.

4. Conclusions

The immiscible PP/NR blends of various concentrations were ultrasonically treated during extrusion in a single screw ultrasonic reactor. Mechanical properties of the treated blends were found to improve significantly in comparison with those of untreated blends. Under most treatment conditions, no significant differences in the viscosity of the treated and untreated blends were observed. The SEM photomicrographs of PP/NR blends showed that after annealing the domain size in the morphology of treated blends was much smaller than that of untreated blends. The AFM studies revealed the development of interfacial roughening and improved interfacial adhesion between PP and NR phases and creation of a thick interfacial layer in the blends subjected to ultrasonic treatment leading to improved mechanical properties. At the same time weak adhesion

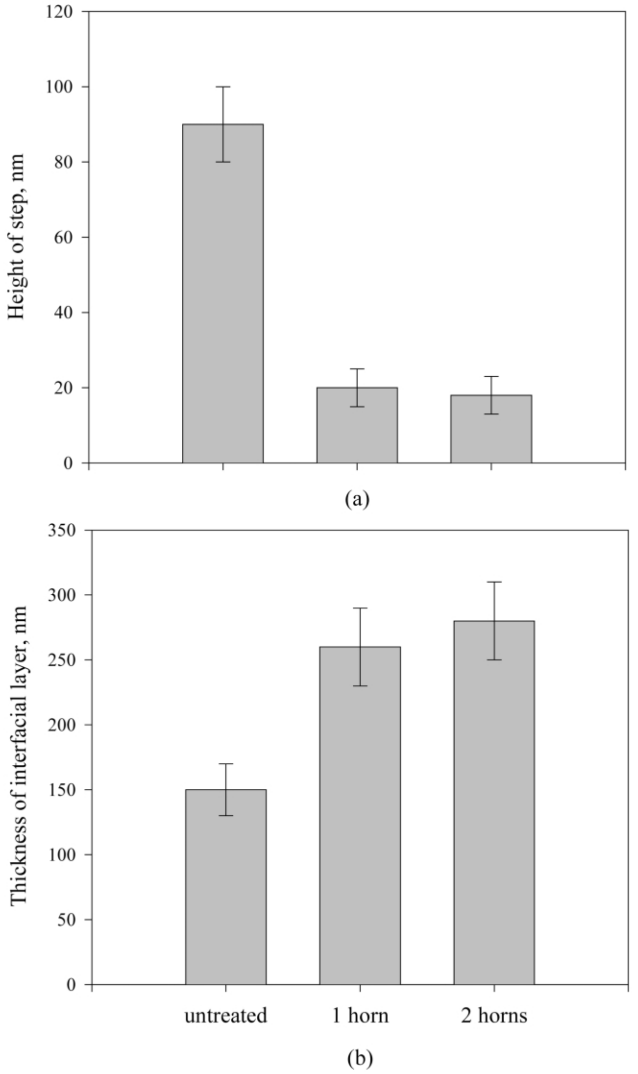


Fig. 17. The height of step at the interface (a) and the thickness of interfacial layer (b) 50/50 PP/NR blends untreated and treated with one horn and two horns at a gap of 4 mm, a flow rate of 0.63 g/s, and an amplitude of 10 μm.

and delamination at the interface were found in the untreated blends. The improved interfacial adhesion, morphology, and mechanical properties are believed to be due to the formation of in situ PP/NR copolymer at the interface of two immiscible polymers caused by an ultrasonic treatment without the use of any chemicals.

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