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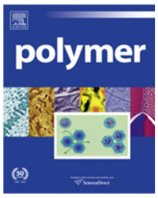

Stabilization and control of phase morphology of PA/SAN blends *via* incorporation of exfoliated clay

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ABSTRACT

The effect of nanoclay on phase morphology development of polyamide/styrene–acrylonitrile (PA/SAN) blends has been investigated. PA/SAN blends of various compositions, with and without the presence of exfoliated nanoclay in the PA phase, were prepared and the morphology of these blends was examined. Efforts have also included the study of morphology stability of these blends and the nanoclay effect on morphology stability of PA/SAN. The results suggest that at compositions where PA remains as the matrix domain, the nanoclay can be effective on reducing dispersed domain size to less than half the original size and furthermore improving the morphology stability of PA/SAN blends upon annealing by preventing coalescence of SAN domain. Implication of the present finding for effective preparation of stable incompatible blends is discussed.

1. Introduction

Polymer blending is an area of study that has been demonstrated to be an effective approach in achieving enhanced material properties, better processability, lowered cost, etc. In order to achieve desirable final properties of polymer blends, it is necessary that the phase morphology of the blends be controlled [1–15]. However, due to the incompatibility of most polymers, the use of polymer blends has been limited and physical or chemical compatibilizers, such as block (or graft) copolymers or in-situ reactive compatibilizers, have been used to reduce domain sizes and enhance interfacial adhesion between the two phases [16–28].

One recent method of controlling and tailoring morphology of polymer blends is the addition of nanoparticles as a compatibilizer into immiscible blends [16,28–34]. The potential use of nano-additives, such as organoclay or silica nanoparticles, in tailoring the morphology of various polymer blends can have a wide range of advantages, such as enhancing material properties, ease of processing, and, especially, lower cost in comparison to copolymer compatibilizers. It should be noted that, because of its physical nature, copolymer compatibilizers could plasticize the matrix and soften the interface, thus compromising physical and mechanical properties of the matrix [11,19–24,35], while on the contrary, nanoparticles, such as clay, have been shown to enhance the mechanical and thermal properties of polymers and stabilize different crystalline phases [36–38].

Some research groups have already shown that organoclay can act as a compatibilizer in immiscible polymer blends [16,39–41]. An addition of organoclay in PPO/PA6 [42], PC/SAN & PMMA/EVA [43], PMMA/PEV [41], PS/PP [44], PA6/EPR [16,45], PA6/PP [46,47], PS/PEMA [48], and PMMA/SAN [31], has been shown to reduce of domain size of the dispersed phase. This effect has been attributed to three possible reasons [16]: (1) the large surface area of organoclay enables formation of *in-situ* grafts during melt processing, enhancing blend compatibility and reducing dispersed phase domain size [40]; (2) addition of organoclay increases the viscosity of the matrix, resulting in reduction of dispersed domain size [49]; (3) the organoclay in the polymer matrix effectively prevent the coalescence of dispersed domains, thus leading to dispersed domain size reduction [16,46,47,49]. Nesterov and Lipatov [39] have shown that the compatibilizing effect of the filler depends on the change in free energy of mixing between the two polymers, especially for immiscible blends. Gelfer et al. [40] and Zhu et al. [50] showed that the addition of organoclay drastically reduced the domain size of PS/PMMA blends. Their explanation was a combination of partial compatibilization by excessive surfactant in organoclay and increased matrix viscosity. Khatua et al. [16] indicated that the addition of a small amount (0.5 wt%) of organoclay in PA6/EPR blends could significantly reduce the domain size when PA6 is the matrix phase, and upon annealing the domain size will not increase noticeably. Thus, organoclay exhibits a compatibilizing effect on these blends. However, when EPR becomes the matrix

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Table 1
MVR and M_W of SAN employed in the study.

System	AN content	MVR @220 °C (cm3/10 Kg)	M_w (g/mol)
SAN	24%	64	137,000

phase, the addition of organoclay shows no effect on the reduction of dispersed domain size. On the other hand, nanoclay addition does not always lead to reduction in domain size and in phase stability. For example, Ferreiro et al. [51] did not observe any compatibilization effect by organoclay in PMMA/PEO blends. Therefore, the subject of compatibilization by organoclay in polymer blends still requires further investigation and clarification.

It should be noted that the majority of the above research efforts involve either the use of organoclay particles without exhibiting full exfoliation or the introduction of significant amount of intercalating agents in the polymer blends. Consequently, complication due to variations in clay particle sizes and intercalating agent type in the polymer blend can lead to ambiguous findings. The recent commercial success of Ube Industries in the *in-situ* preparation of well-exfoliated clay in nylon has made it possible to study how exfoliated clay particles alone can serve as compatibilizer for immiscible blends. This will enable better fundamental understanding on the roles the clay particles play to disperse and stabilize the phase morphology of immiscible blends.

In this research, the effect of nanoclay in PA matrix on the morphology and dispersion of SAN phase will be investigated. Their phase stability up to 10 min of annealing at 260 °C is also investigated. Possible physics responsible for the phase break-up and stability of the domains is discussed.

2. Experimental

2.1. Materials

SAN material (VLN Grade) for this study was prepared and characterized for molecular weights and melt viscosity by BASF (Table 1). A pair of PA systems, neat (1022B grade) and PA/clay nanocomposite containing 2 wt% of exfoliated nanoclay (1022C2 grade), was obtained from Ube Inc. All materials were received in pellet form. The molecular weights and melt volume rate (MVR) of the materials are given in Table 2.

2.2. Blending

PA was dried at 110 °C for 3 h prior to blending. Mixing was performed at 260 °C for 7 min at 30 rpm using a Haake mixer (System 40). Blends were prepared at various compositions (Table 3). Prefixes **N** and **C** have been given to the systems containing neat PA and PA/clay nanocomposite, respectively. The systems have been named in a way consisting of two numbers: the first and second indicating the amount (wt%) of PA and SAN, respectively.

After the mixing stage, in order to investigate the blend morphology stability, the blends were quenched under various thermal conditions: (A) quenched in ice water immediately after blending, (B) and (C) were kept at processing temperature of 260 °C and then quenched after 3 and 10 min, respectively.

Table 2
MVR, M_W, and T_m of PA systems employed in the study.

System	Mn (g/mol)	Melting Temperature (°C)	Melt Viscosity@250°C shear rate = 1000 s^{-1} (Pa s)
PA	22,000	220	280
PA/Clay	22,000	220	300

Table 3
Blend compositions.

System	PA content	SAN content
N80/20	N-80%	SAN-20%
N60/40	N-60%	SAN-40%
N40/60	N-40%	SAN-60%
N20/80	N-20%	SAN-80%
C80/20	C-80%	SAN-20%
C60/40	C-60%	SAN-40%
C40/60	C-40%	SAN-60%
C20/80	C-20%	SAN-80%

2.3. Morphology observation

After preparation, the blend morphologies were studied to investigate the effect of nanoclay upon blend morphology and its stability. Blend samples were extracted from the Haake mixer and quenched, followed by cutting and polishing thoroughly on one side. In case of systems with PA as the matrix phase and SAN as the dispersed phase, the sample surfaces were etched in THF for 24 h to remove the SAN phase on the surface. In the case of systems with SAN as the matrix phase and PA as the dispersed phase, the sample surfaces were etched in formic acid for 2 h to remove the PA phase from the surface. Samples were then coated with a thin layer of Au/Pd with an approximate thickness of 30–40 Å using a Cressington 308 coater to prevent charging, and finally examined using a JEOL-6400 Scanning Electron Microscope (SEM).

For transmission electron microscopic (TEM) observation, the thin-section samples extracted from the core region of the specimens were prepared by microtoming (Ultracut E) and the thin-sections having 60–80 nm in thickness were deposited on carbon coated Cu grids. Sample grids were examined on a JEOL 1200 EX electron microscope operated at an accelerating voltage of 100 kV. Complete details of the microtomy and microscopy techniques employed here are given in prior publications [52–56].

2.4. Rheological measurements

To investigate the rheological behavior of the blends, dynamic viscosities of the samples were obtained using a TA instruments (ARES-G2) with 25 mm diameter parallel plates. Samples of about 1 g were subjected to a frequency sweep at 260 °C under a nitrogen environment to prevent degradation. The strain amplitude (γ_0) was set at 1% which was shown to lie within the linear viscoelastic region through an amplitude sweep at 260 °C.

2.5. Particle size measurements

In order to determine the extent of dispersion of the blend components, SEM micrographs of selected systems were examined using image-j analysis. Based on the particle size results, the particle diameter was calculated. The averages of particle diameter have been listed in Table 4.

Table 4
Average particle diameter (μm) of selected systems.

System	A	B	C
N-60/40	1.07 ± 0.69	1.95 ± 1.02	3.02 ± 1.57
C-60/40	0.63 ± 0.29	0.71 ± 0.42	0.89 ± 0.41
N-80/20	0.48 ± 0.18	0.67 ± 0.45	0.90 ± 0.64
C-80/20	0.18 ± 0.14	0.20 ± 0.17	0.26 ± 0.20

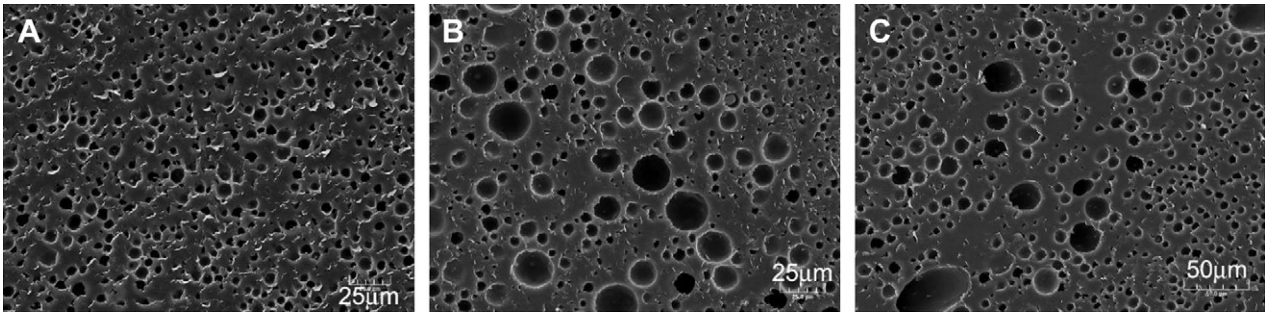

Fig. 1. Morphology of system N60/40-A, B, and C.

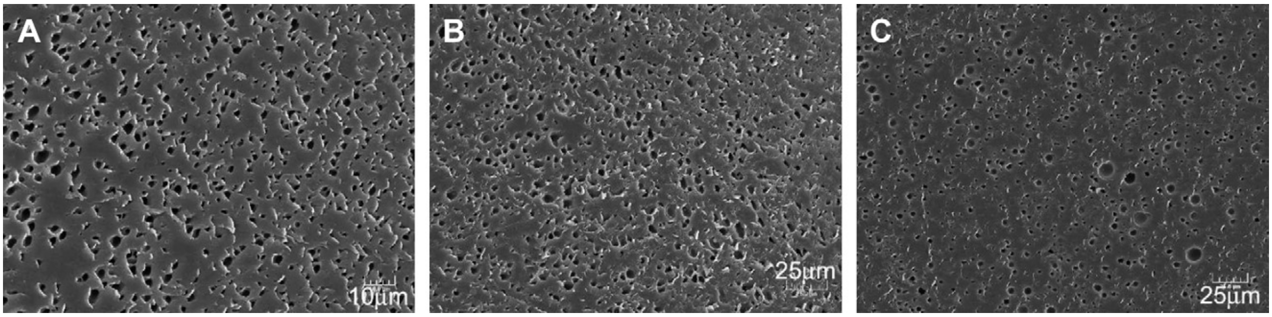

Fig. 2. Morphology of system C60/40-A, B, and C.

3. Results and discussion

The good quality exfoliation and dispersion of nanoclay in the Ube nylon matrix have been demonstrated and reported elsewhere [57]; it will not be elaborated here. SEM micrographs of both N and C systems with 60/40 and 80/20 compositions are shown in Figs. 1–4. In both of these compositions due to the relative high viscosity of PA against SAN as well as the composition of the phases, the PA component is the matrix phase.

The SEM micrographs in Figs. 1–4 indicate that the relative domain size of the dispersed phase varies significantly among the systems. Comparison of the relative domain sizes in Figs. 1(a) and 2 (a) suggests that in the case of clay containing systems, the average domain size is approximately half of that in the neat nylon systems. This effect on the blends morphology is related to the presence of nanoclay in the nylon matrix and the compatibilizing effect that clay could have on these blends [16,39].

Furthermore, comparisons of Fig. 1(b) and (c) with Fig. 2(b) and (c), respectively, show a much more significant difference in the observed domain sizes. Considering the fact that these systems were quenched after 3 and 10 min of thermal aging, respectively, the growth rate of domain size can be a relative indicator of how stable the blend morphology is within the system. It is shown that the growth rate of domain size for clay containing systems is significantly lower than that of neat PA-based systems. This indicates that the presence of nanoclay in the PA matrix has not only resulted in better dispersion and decrease in the SAN domain size but also it has notably stabilized the blend morphology, limiting diffusion and coalescence of SAN domains towards each other. The average particle diameter of these systems has been listed below in Table 4.

To explain the above observed reduction in domain size and increase in morphology stability, it is thought that the nanoclay acts as a physical barrier in the matrix phase, which help limiting and preventing the diffusion and coalescence of the dispersed SAN particles towards each other. This finding is in agreement with previous efforts explaining the increase in morphology stability with the introduction of organoclay in PA6/EPR blends [16] and various other systems [16,43,49].

The locations in which the nanoclay resides in the blends can have a significant effect upon the blend morphology and therefore must be determined. To do so, TEM was performed on the C80/20

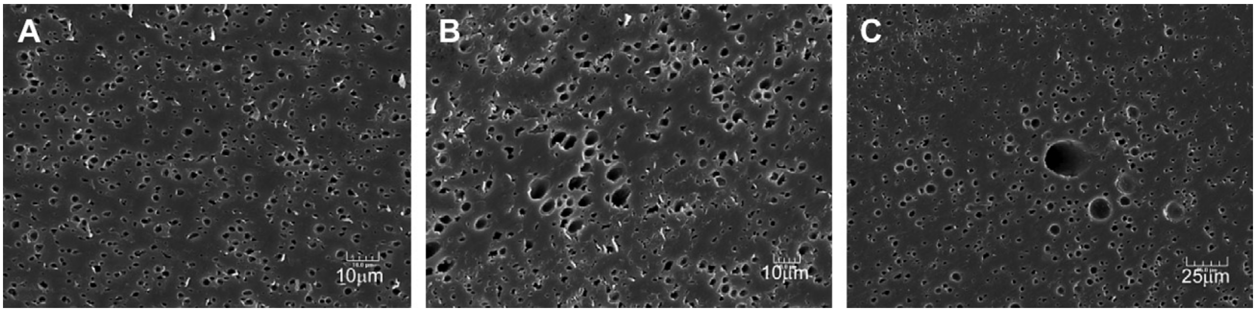

Fig. 3. Morphology of system N80/20-A, B, and C.

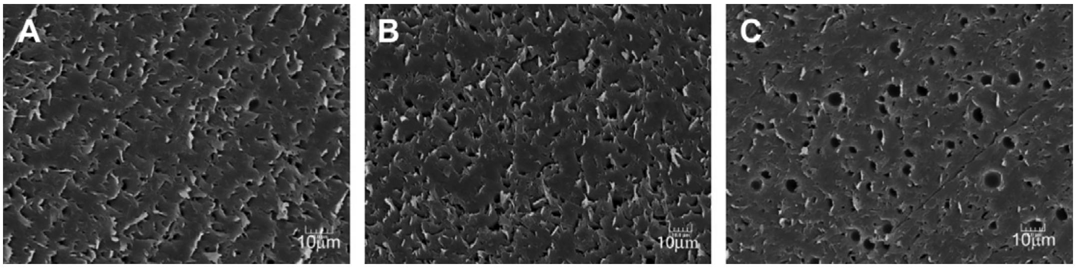

Fig. 4. Morphology of system C80/20-A, B, and C.

and C60/40 systems (Figs. 5 and 6). In these images, it can be seen that nanoclay platelets exist only within the PA matrix. It can also be seen that the platelets are well dispersed and do not form any detectable aggregation, which is consistent with previous report of showing complete exfoliation of nanoplatelets in PA [57]. The presence of exfoliated nanoplatelets within the matrix can affect the blend morphology either by enhancing the compatibility between the two phases [40,43,49] or by altering the matrix viscosity, thus limiting the coalescence of the dispersed domain towards each other [29,31,46,48].

During melt mixing, the morphology development is mainly determined by a flow-induced process [42]. During this process the dispersed domain particles deform, break-up, coalesce, and finally reach a dynamic equilibrium state [42,58,59]. The high aspect ratio exfoliated clay platelets can act as an effective barrier during the coalescence step of the processing. Based on the TEM images shown in Figs. 5 and 6, the clay platelets only exist within the PA phase [16,41,60]. PA is generally more polar compared to SAN and thus clay platelets prefer to remain within the PA matrix rather than to migrate into SAN phase.

Based on the above findings, a schematic showing the role of clay platelets in PA matrix can be seen in Fig. 7. Since the exfoliated platelets exist in the PA phase, they can act as a physical barrier to limit the coalescence of the SAN dispersed phase. Furthermore, the effectiveness of the clay in improving morphology stability that can be seen in comparison of various quenching times (A, B, C) can also be explained by this mechanism.

To investigate the role of viscosity changes in the observed morphology evolutions, dynamic viscosity curves of the PA, PA/clay, and SAN systems are shown in Fig. 8. It can be seen that the addition of clay to the PA phase has resulted in a slight increase of melt viscosity measured at processing temperature of 260 °C. Taking into consideration the shear rate induced by the Haake mixer, which is estimated to range from 8.7 s^{-1} (deep section of

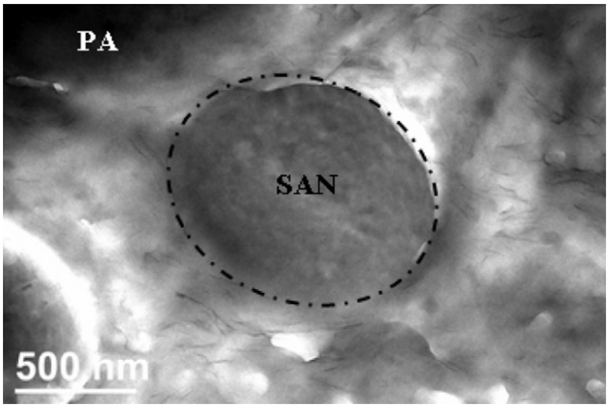

Fig. 5. TEM micrograph of C60/40-A.

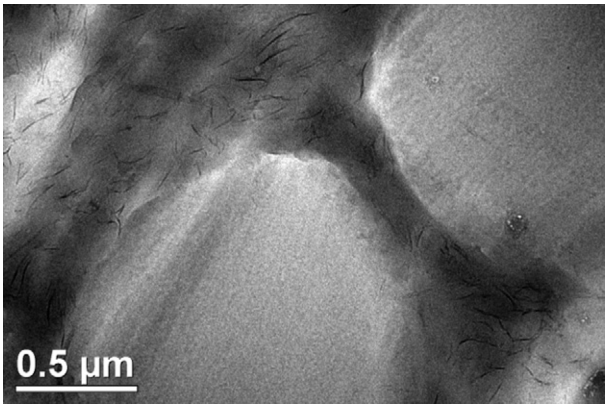

Fig. 6. TEM micrograph of C80/20-A.

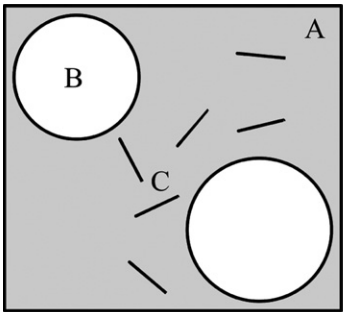

Fig. 7. Schematic of PA matrix (A) and SAN (B) domains depicting the role of clay platelets (C) in preventing coalescence of SAN phase.

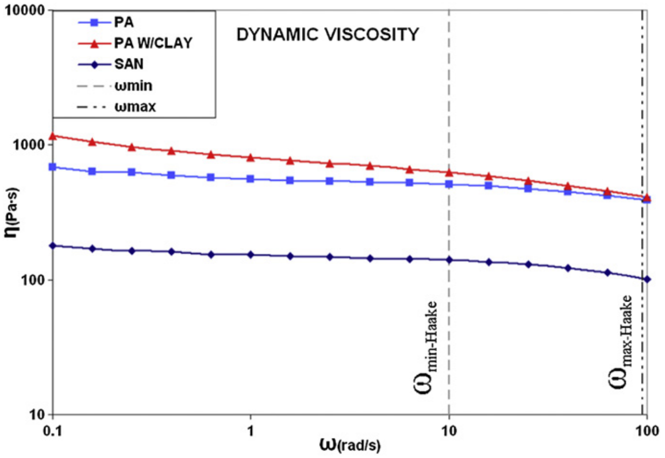

Fig. 8. Dynamic viscosity curves of PA, PA w/clay, and SAN.

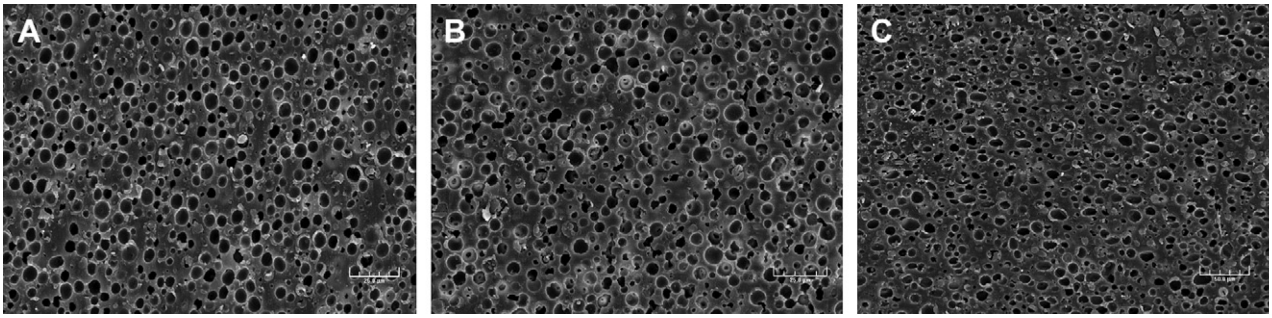

Fig. 9. Morphology of system N40/60-A, B, and C.

mixer) to a maximum of 34.2 s^{-1} (mixer's gap) [61–64], the increase in PA viscosity due to clay addition is found to be minimal (Fig. 8). However, owing to the significant differences in viscosity between the PA and SAN phases and the fact that PA is the more viscous phase, the slight increase in melt viscosity of PA/clay phase cannot contribute to the observed significant reduction of average dispersed domain size of SAN [16,65]. Furthermore, since the clay resides in the PA phase, the clay particles cannot effectively serve as defects to facilitate break-up of the SAN phase. Consequently, the clay particles in the PA phase only serve as a barrier to delay and minimize phase coalescence. Thus, the phenomenon from which the clay exhibits a compatibilizing effect in PA/SAN blends is of kinetic nature.

The possibility of clay platelets acting as a thermodynamic compatibilizer with respect to the blend components must also be taken into consideration [66]. The introduction of nanoclay into the blend as a third component leads to changes in the free energy of mixing [67]. The transient occurrence of exfoliated clay platelets along the PA/SAN interface can reduce the interfacial tension and therefore enhance the wetting between the two phases. This, in turn, is known to result in the reduction of average dispersed domain size [49,67,68]. Therefore, the addition of nanoclay may contribute to the compatibility of PA and SAN components in both thermodynamic and kinetic aspects. These mechanisms can act in parallel and even synergistically [36,67]. However, the contribution of the thermodynamic compatibilizing effect of clay may be limited mainly due to the fact that the effectiveness based on this method depends on the affinity of the clay with each of the PA and SAN components as well as the preference of clay location in the vicinity of the interface [42]. In this study, since clay only shows affinity to PA phase, the presence of clay in the PA phase may indeed both physically and thermodynamically repel the adjacent SAN domains from coalescing. In this regard, exfoliated clay would be highly beneficial. This is probably the reason why only 2 wt% of clay can exert such a significant effect on the phase stability of SAN phase in PA matrix. Another mechanism which must be taken into consideration is the possible partial adsorption of some SAN molecules on clay surfaces. This mechanism which has previously been observed in various studies [58] may render limited compatibility between the components and act as a physical compatibilizer.

In order to investigate the efficiency of these exfoliated clay platelets on the PA/SAN blend morphology, comparison can be drawn to previous study by Khatua et al. [16], which indicated that in PA/EPR blends when PA is the matrix phase the incorporation of nanoclay into the blend will result in size reduction of EPR domains to 30% of the original size. Furthermore, in these systems upon annealing the relative domain size of the EPR domains increased by 40% and 200% in the presence and without the nanoclay platelets respectively. In the current study, the incorporation of the nanoclay has reduced the rate of domain size growth upon annealing from 200% without the nanoclay to approximately 30% in the presence of the nanoclay while PA is the matrix phase. It should be noted that the amount of nanoclay used in both scenarios were similar.

For blends with SAN as the matrix phase, the presence of clay in PA domains appears not to affect phase morphology and phase stability. SEM images of systems with N40/60C40/60, N20/80, and C20/80 compositions are shown in Figs. 9–12.

The SEM images of the various systems shown above indicate that the blends all exhibit PA as the dispersed phase. In this case, there is no noticeable difference between the average domain size of the dispersed phase between systems with and without the presence of nanoclay. Furthermore, comparisons of the systems quenched after 3 and 10 min with or without the presence of nanoclay indicate that, likewise, the addition of nanoclay has not been effective on improving the morphology stability of these blends.

The TEM micrographs of C20/80 and C40/60 systems are shown in Figs. 13 and 14. It can be seen that in these systems, which have SAN as matrix, the nanoclay platelets are located entirely inside the dispersed PA phase. Therefore, the ineffectiveness of nanoclay on

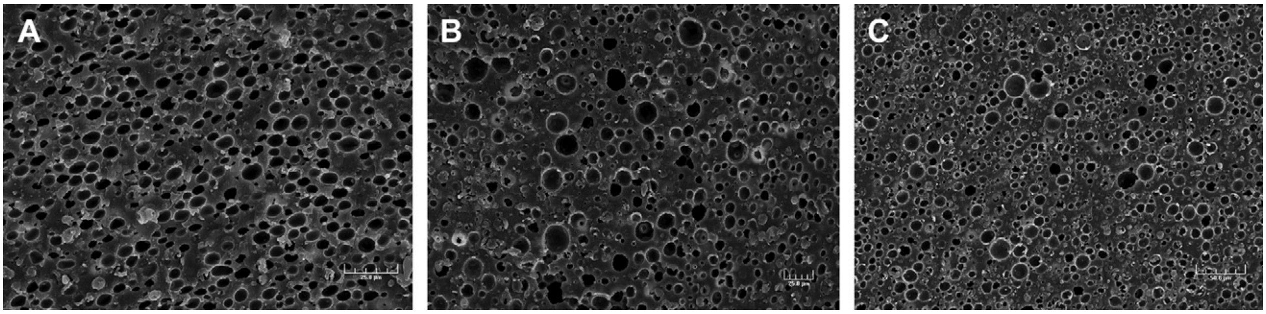

Fig. 10. Morphology of system C40/60-A, B, and C.

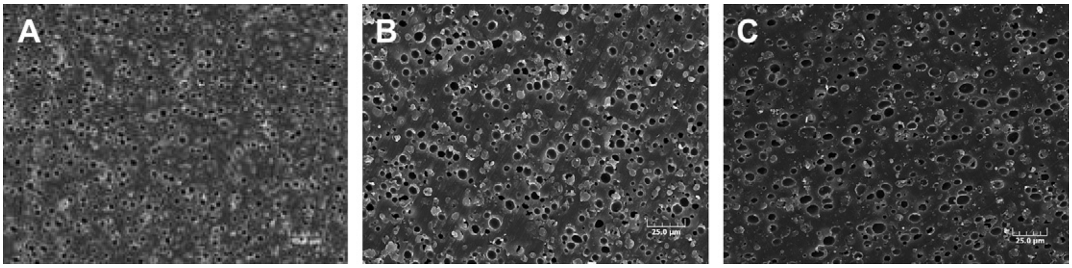

Fig. 11. Morphology of system N20/80-A, B, and C.

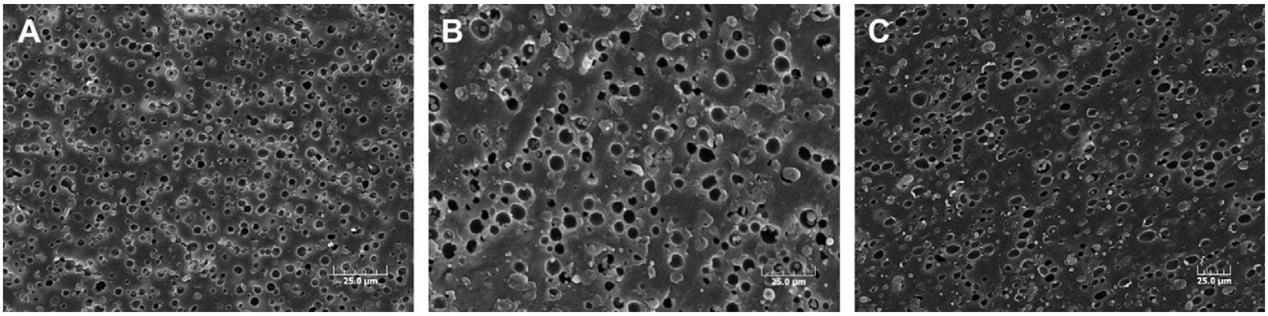

Fig. 12. Morphology of system C20/80-A, B, and C.

the morphology stability can be due to the fact that the nanofiller is mainly located inside the dispersed phase in this case. This in turn has limited the ability of clay to influence phase domain sizes during mixing. These results are also in agreement with previous findings by Khatua et al. [16], which showed that in PA6/EPR blends where EPR is the matrix phase, addition of organoclay did not result in significant morphological changes.

Based on the above findings, the thermodynamic compatibilizing effects of the clay platelets on PA/SAN blends can be objectively taken into consideration. In the case that the SAN is the matrix component, the addition of clay to the blends does not result in any significant changes in the average dispersed domain size. Furthermore, evidence of TEM micrographs in Figs. 5,6,13 and 14 indicate that clay platelets do not show preference to reside at or near the interface between the two phases. Therefore, it can be concluded that the compatibilizing effect of the nanoclay on PA/SAN blends is more of kinetic origin. This kinetic compatibilizing effect of clay on PA/SAN blends with PA as matrix can play a significant role in various applications where small SAN domain size is preferred.

In this study, exfoliated clay in the PA matrix of the PA/SAN blends can greatly reduce the average dispersed domain size, which has also been claimed in other cases where the clay is not fully exfoliated [47,69]. Furthermore, the effectiveness of utilizing exfoliated clay to improve the morphology stability with respect to thermal aging is emphasized and is in agreement with previous claims [16,36]. In other words, the exfoliated clay platelets in the matrix phase of PA/SAN blends are effective in preventing particle coalescence, rendering the blend morphology more stable. In this case, the incorporation of 2 wt% of exfoliated clay leads to a dispersed domain size increases of less than 50% at processing temperature of 260 °C for 10 min. Based on the extent of phase separation observed in the absence of clay which led to increases up to approximately 200% in particle size, it can be concluded that the rate of SAN particle coalescence has been significantly reduced in the presence of exfoliated clay platelets in the matrix.

Finally, the utilization of exfoliated clay in the matrix phase of a blend can prove to be advantageous. Clay dispersion in a polymer blend has been shown to influence viscosity, interfacial tension,

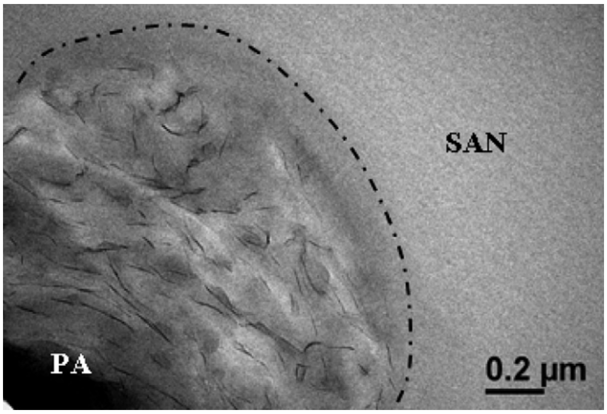

Fig. 13. TEM of C20/80-A.

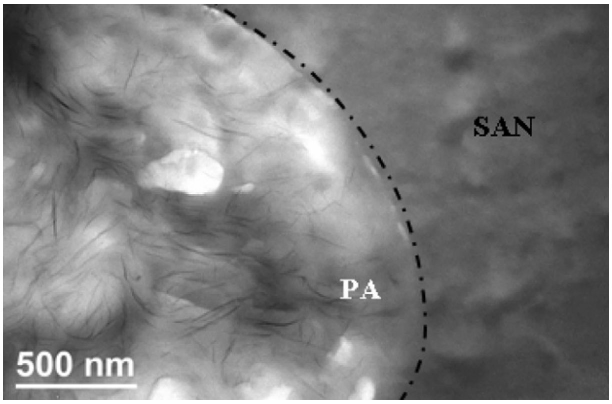

Fig. 14. TEM of C40/60-A.

mechanical properties, barrier properties, etc. The influence of clay on physical and mechanical properties of PA/SAN blends will be examined in future studies.

4. Conclusion

Nanoclay in PA is shown to affect the morphology of PA/SAN blends when PA is the matrix phase. The presence of nanoclay in PA has been shown to be effective in both reducing the average size of the SAN dispersed domain and, more importantly, significantly stabilizing the morphology of these blends. However, at compositions where PA is the dispersed phase, due to the reason that all nanoclay platelets exist inside the dispersed PA domain, the presence of the nanoclay has little or no effect upon the blends morphology or their phase stability at elevated temperatures. Furthermore, it is seen that the pre-existing exfoliated clay within the PA matrix of PA/SAN blends can be more effective upon stabilizing the blends morphology in comparison with intercalated clay containing systems.

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