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The gelation behaviors of the reactive blends of nylon1212 and functional elastomer

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Abstract Studies on the gelation behaviors of the reactive blends of nylon1212 and functional elastomer were carried out. The results show that the curves of the storage modulus(G')–frequency (ω) exhibit a gel plateau in the low ω region, and the transition from liquid-like to solid-like viscoelastic behaviors emerges with the concentration of SEBS-g-MA increasing. There exist the gelation behaviors in the blending process similar to those of crosslinking polymer. Based on Winter's method, the gel point of blends is determined to be, $\varphi_g = 17.45$ wt%, and the corresponding value of tanδ is 1.44. The gel index n calculated is 0.61 and the gel strength S_g is 1.08×10^4 Pa s$^{0.61}$. However, the non-reactive blends of nylon1212 and elastomer have no emergence of gelation behaviors. The morphology analysis shows that the gel point for the reactive blends is a threshold of cocontinuous morphology, and morphology analysis can also be a method to determine the gel point.

Introduction

As is well-known, the chemical gel point is reached when the largest molecule or super molecular cluster diverges to infinity, and physical gel point is reached when polymers lose their chain mobility and have a sharp transition from liquid-like behavior to solid-like behavior. This critical point maybe a critical time, temperature, or concentration of polymers, in which the viscoelastic parameters of systems, zero shear viscosity, and relaxation time have some deviation [1–5]. Though there are many methods to determine the gel point, the method based on zero shear viscosity (η_0) and equilibrium modulus (G_e) is a popular one [6–14]. In the gel point, $\eta_0 \rightarrow \infty$ and $G_e = 0$. However, it is difficult to measure η_0 and G_e near the gel point. Firstly, measurement of shear viscosity is usually transient mode and measure time is relatively short. The sample is very difficult to reach the steady state because the relaxation time is long near the gel point. Secondly, shear can destroy the network structure and postpone the emergence of gel point. The phase separation and the glass transition also can make $\eta_0 \rightarrow \infty$.

Tung and Dynes provided that G' should be equivalent to G'' in the gel point, but they also found that the crossovers of G' and G'' depend on the frequency, which is inconsistent with the theory that the gel point is independent of the frequency [15]. Winter et al. considered that the crossover of G' and G'' corresponds to the gel point only for the system for which the relaxation index is 0.5 [16, 17]. However, for most network systems, though stress relaxation at gel point follows a power law, the relaxation exponent n is not 0.5. Gel point clearly does not coincide with the G' and G'' crossover in such cases.

Usually, the relaxation spectrum of gel can be expressed as

$$H(\lambda) = \frac{S_g}{\Gamma(n)} \lambda^{-n} \tag{1}$$

In the terminal region, the relaxation modulus G(t) is

$$G(t) = S_g t^{-n} \tag{2}$$

in which $\Gamma(n)$ is the gamma function. S_g, the strength of the gel related to the mobility of the crosslinking chain

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segments, is only a material parameter that depends on the flexibility of molecular chains and the crosslinking density at gel point. The relaxation exponent n (physically restricted for $0 < n < 1$) is related to the geometry of clusters existing at the gel point, and is a viscoelastic parameter that is related to the cluster size of the gel [18–24].

The shear stress of gel near the gel point can be expressed as [6, 18],

$$\sigma_{21}(t) = -S_g \int_{-\infty}^{t} (t - t')^{-m} \dot{\gamma}(t, t') \mathrm{d}t' \tag{3}$$

If the shear strain is a sinusoidal function in the dynamic experiment, the amplitude of shear strain is γ_0, and shear strain γ(t) can be expressed as

$$\gamma(t) = \gamma_0 \sin(\omega t) \tag{4}$$

thus, Eq. 3 can be represented as

$$\sigma_{21}(t) = G'(\omega)\gamma_0 \sin(\omega t) + G''(\omega)\gamma_0 \cos(\omega t) \tag{5}$$

and the frequency dependence of the dynamic shear modulus at gel point is deduced to be

$$G'(\omega) = G''(\omega)/\tan(n\pi/2) = \Gamma(1 - n)\cos(n\pi/2) S_g \omega^n \tag{6}$$

Therefore, the loss tangent tanδ becomes independent of frequency in the gel point

$$\tan\delta = G''/G' = \tan(n\pi/2) \tag{7}$$

From Eq. 7, it can be concluded that n evaluates the difference between the viscoelastic system and viscous liquid or elastic solid in rheology. It can also be noted that if elastic network forms in the gel point, n is impossibly equal to 1. Usually, the relatively low n implies that the system has high elasticity.

In recent two decades, a lot of researches concerning nylon/functional elastomer blends have mostly involved in the aspects of mechanical and thermal properties [25–29]. To our knowledge, a few reports have been dealt with rheological behaviors of their melts [30–32]. It is noted that the rheological measurements not only give much useful information on material molding and processing, but also provide an effective approach to characterize of structure and properties of polymer materials [33, 34]. The emergence of gelation process exhibits that the crosslinking density increases or a kind of new special structures (such as hydrogen bond, supermolecule structure) forms, which is bound to change obviously the mechanical properties of blends.

Although extensive studies of rheological study on structural transition of crosslinking polymers were carried out, such as epoxy [35–38] and dicyanate resin [39], poly(vinyl alcohol) [22], and polybutadiene [23], few researches reported the structural transition of reactive blend system. This work presents the study of gelation behavior of nylon1212/SEBS-g-MA reactive blends by using rheological measurements and morphological analysis, and compares this particular behavior with that of non-reactive blends of nylon1212/SEEPS blends.

Experimental

Materials

Nylon1212 (melt flow index = 22.43 g/min under 5.0 kg pressure at 230 °C, melting point is 184 °C, relative density is 1.01) used here is a product from Shandong Dongchen Engineering Plastic CO., LTD, China. The maleated triblock copolymer styrene-b-(ethylene-*co*-butylenes)-b-styrene (SEBS-g-MA, Kraton FG 1901) is a product of Shell Chemical Co., USA, which contains 29 wt% styrene, and molecular weight of PS and poly (ethylene-*co*-butene) copolymer blocks is about 7,500 and 37,500. The weight percent of MA grafted onto this elastomer is 1.7–2.0 wt%. Styrene-[ethylene-(ethylene-propylene)]-styrene block copolymer (SEEPS 4033, polystyrene end-blocks = 30 wt%, density = 0.91 g/cm^3. Melt flow index $\leq$ 0.1 under 2.16 kg pressure at 230 °C, viscosity = 50 mPa s in the 10 wt% toluene solution, molecular weight of PS and poly (ethylene-(ethylene-propylene)) copolymer blocks is about 10,000 and 46,900) is a product of Kuraray Co., Japan. The antioxidant (B215, relative molecular weight = 647, T_m = 453–458 K) is a product of Ciba-Geigy Co., Switzerland.

Preparation of samples

According to the different ratios, nylon1212 and SEBS-g-MA and 1 wt% antioxidant were blended in a Haake torque rheometer (Rheoflixer Polylab) at 190 °C for 10 min. The samples were compressedly molded at 190 °C into disks of 25 mm in diameter and 1.2 mm in thickness. The samples of nylon1212 and SEEPS blends were prepared in the same way.

Rheological measurements

Melt rheological tests were conducted on an ARES Rheometer (Rheometrics Inc. USA) in parallel plates oscillatory mode. The dynamic frequency (ω) sweep was performed from 0.01585 to 100 rad/s, and the strain amplitude was maintained as 1% in order to ensure that rheological behavior is located in the linear viscoelastic region. The temperature of all the tests was maintained at 190 °C.

Morphological observation

The morphologies of the blends were observed using a JSM-5510LV scanning electron microscope (SEM). All samples were fractured in liquid N_2 and etched in boiling xylene for 12 h for removing elastomeric phase of the blends. The etched surface after properly drying was coated with a conductive gold layer before SEM analysis.

Results and discussion

Gelation behaviors of reactive blends

Figure 1 shows G' and G'' varying with ω for nylon1212/SEBS-g-MA blends containing different concentration of functional elastomer (φ). The viscoelastic behaviors of virgin nylon1212 and SEBS-g-MA are obviously different. The viscoelastic behaviors of blends depend on the component of blends. The virgin nylon1212 seems to exhibit the liquid-like viscoelastic behavior, $G' \propto \omega^2$, $G'' \propto \omega$ at low ω, while the blends do not. With φ increasing, the dependence of G' and G'' of the blends on ω becomes weak. When φ is larger than 20 wt%, G' and G'' of the blends almost show no dependence on ω at terminal region and exhibit the solid-like viscoelastic behaviors. It is a symbol for emergence of gel plateau according to the results of Nijenhuis and Winter [40]. At the low ω region, with φ increasing, the slopes of G'–ω curves change from 2 to 0 and the slopes of G''–ω curves change from 1 to 0. Therefore, when the slopes of G' and G'' curves attain a given value, the transition from liquid to solid emerges. The corresponding φ should be the gel point, which is located in concentration of SEBS-g-MA between 0 and 20 wt%.

Figure 2 presents the relationship between loss tangent (tanδ) and ω for nylon1212/SEBS-g-MA blends. It can be observed that the curve of pure nylon1212 is almost a straight line, and tanδ is proportional to ω^{-1} in the whole ω region, exhibiting the rheological behavior of homogeneous polymer [41]. The curve of SEBS-g-MA shows a peak and a valley, corresponding to the micro-separation structure of block copolymer. The curves of the blends with high concentration of elastomer show the same shape as that of pure functional elastomer, which demonstrates that a special structure is formed in the blend process. However, it is noted that the shapes of tanδ curves vary with the increase in concentration of SEBS-g-MA. The value of tanδ is over 1.0 for blend with 10 wt% SEBS-g-MA, while those of other blends are below 1.0. The change of tanδ value demonstrates that there exist the gelation behaviors in the blending process similar to those of crosslinking polymers. Thus, it can be inferred from Fig. 2 that the gel point is near to 20 wt%.

Determination of gel point

In order to explore further the gelation process, the data of tanδ can be replotted according to the method of Winter.

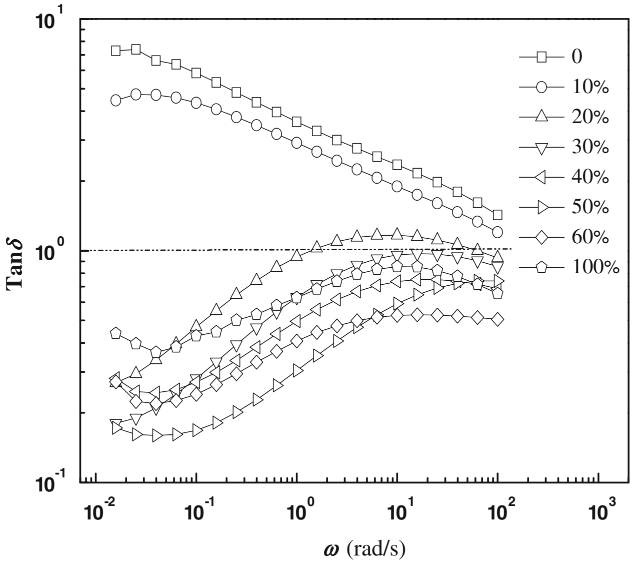
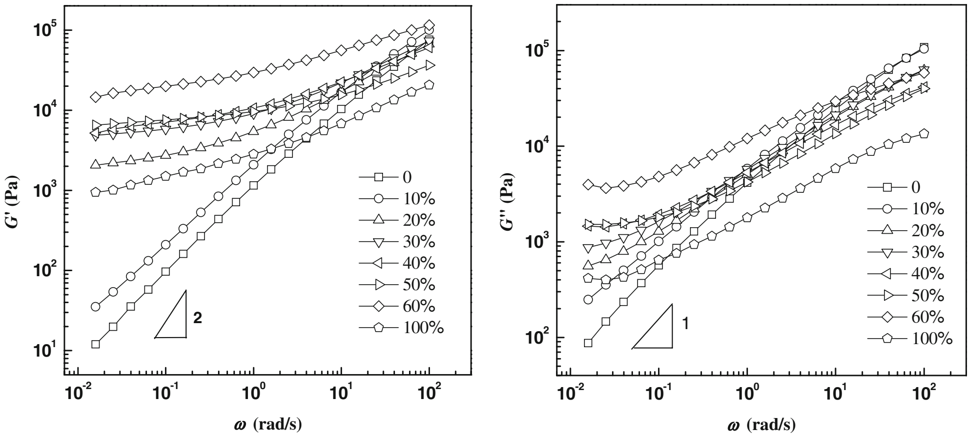


Fig. 2 The relationship between loss tangent (tanδ) and frequency (ω) for nylon1212/SEBS-g-MA blends

Fig. 1 The relationship between the dynamic storage modulus (G') and loss storage modulus (G'') and frequency for nylon1212/SEBS-g-MA blends



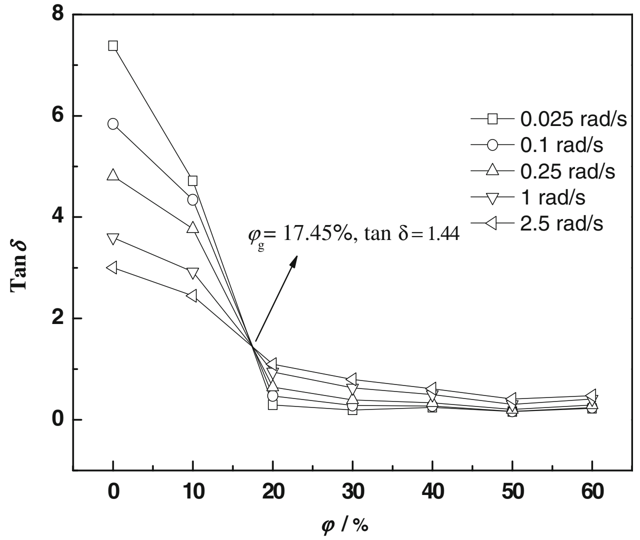


Fig. 3 The relationship between loss tangent tanδ and SEBS-g-MA concentration φ at various angular frequencies for nylon1212/SEBS-g-MA blends

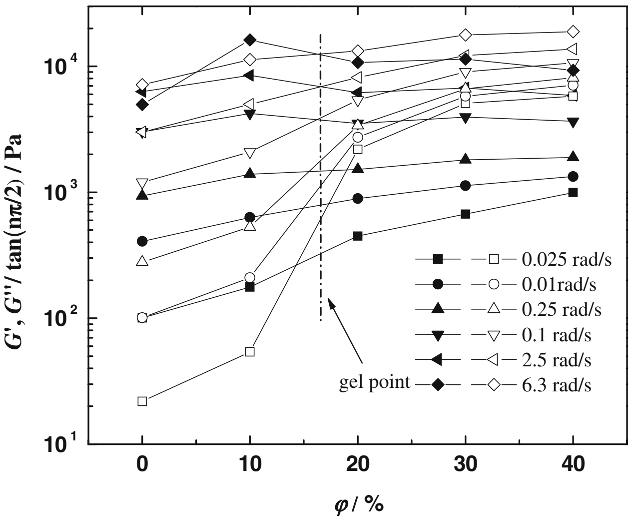


Fig. 4 The relationship between G' (open) and $G''/\tan(n\pi/2)$ (solid) and SEBS-g-MA concentration φ at various angular frequencies for nylon1212/SEBS-g-MA blends. $n = 0.61$ was used to calculate $G''/\tan(n\pi/2)$

Figure 3 shows tanδ as a function of concentration of SEBS-g-MA at different ω for nylon1212/SEBS-g-MA blends. It can be seen that the different curves of tanδ intersect at a constant concentration of SEBS-g-MA, and tanδ is independent of ω at this point. Therefore, we can determine the gel point of blends to be $\varphi_g = 17.45$ wt%, and the corresponding value of tanδ is 1.44. Based on Eq. 7, it can be calculated that the gel index n is 0.61. Many researches demonstrate that n is different for different gel systems. n of chemical gel is 0.5–1 [6, 18, 42], 0.2–0.7 [20], 0.31–0.91 [21], 0.45–0.65 [22], 0.45–0.5 [23], but n of physical gel is relatively large, such as 0.77–0.83 [40], 0.78 [43], 0.62 [44], which coincide with the results of theoretical predictions. n of chemical gel decreases with the increase of crosslinking degree, but n of physical gel does not vary with the concentration. However, it should be noted that it is not enough to investigate the relationship between n and gel structure.

At the gel point, the physical property of gel can be expressed by the gel strength (S_g). When the crosslinking densities of chemical gel increase, n decreases but S_g increases, implying that S_g is related to physical strength of gel network [21, 22]. However, it is difficult to acquire the gel system at gel point and measure S_g directly. It can be seen from Eq. 6 that $G'(\omega)$ and $G''(\omega)/\tan(n\pi/2)$ have an intersect at a concentration, corresponding to the gel point. Therefore, the values of $G'(\omega)$ can be acquired through plotting $G''(\omega)/\tan(n\pi/2)$ with φ at the gel point and then S_g can be calculated.

Figure 4 gives the relationship between $G'(\omega)$ and $G''(\omega)/\tan(n\pi/2)$ and φ at different ω. It can be observed that two curves at the same ω have an intersect, which coincide with the gel point acquired through the independence of tanδ on ω. Furthermore, based on the values of $G'(\omega)$ and Eq. 6, S_g is calculated to be 1.08×10^4 Pa $s^{0.61}$. It is noted that S_g should be the same in a range of the experimental errors but we always choose the average value of S_g corresponding to different ω.

Although the formation and structure characterization of gel have been investigated broadly, the reasons for formation of gel have no popular viewpoint. In general, it can be considered that gel form accompanying emergence of crosslink, phase separation, crystallization, and chain entanglement. As for blends of nylon1212/SEBS-g-MA, two components have an imidization reaction in the blending process and form graft copolymers, which can enlarge the length of macromolecule chain and increase the entanglement density [45, 46]. It can be speculated that the formation of gel for theses blends is caused by the formation of dynamic crosslinking network. However, it is noted that the conditions discussed here are isothermal; when the temperature of measurement change, the location of gel point is bound to shift, because the entanglement density has same degree of dependence on temperature.

Viscoelastic behavior of non-reactive blends

Figure 5 presents the relationship between tanδ and ω for nylon1212/SEEPS blends. It can be seen that the curve of pure SEEPS shows a peak in the low ω region, which is different compared with that of pure SEBS-g-MA. The viscoelastic behaviors of polymers depend on the molecular weight for the same kind of polymers. Here, although SEEPS and SEBS-g-MA elastomers have similar micro-separation

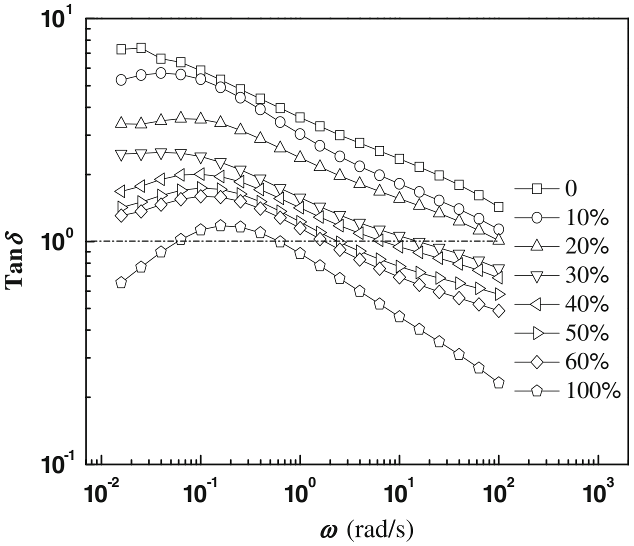


Fig. 5 The relationship between loss tangent (tanδ) and frequency (ω) for nylon1212/SEEPS blends

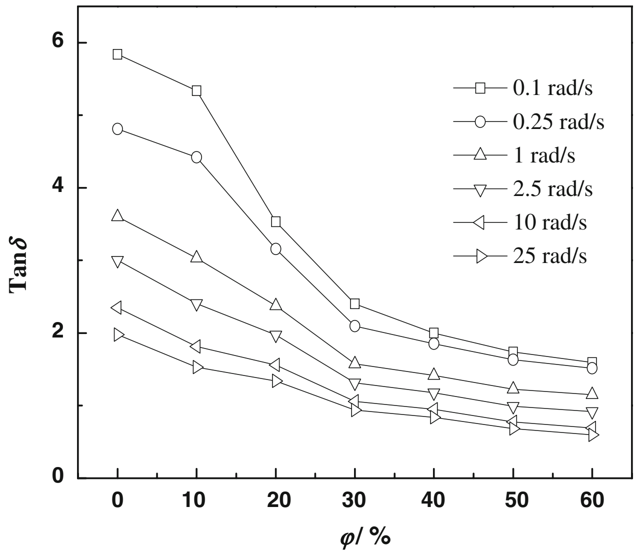


Fig. 6 The relationship between loss tangent tanδ and SEEPS concentration φ at various angular frequencies for nylon1212/SEEPS blends

structures, their molecular weights are different. Different molecular weights make both elastomers show the different shape of tanδ curves. There is no emergence of gelation process similar to those of crosslinking polymers for these blends. It is well known that nylon1212 and SEEPS elastomer are thermodynamic incompatible systems, and there exists poor interaction in both components. Both components form individual phases, and introduction of SEEPS cannot change obviously the length of macromolecular chains and the condition of entanglement. On the other hand, the values of tanδ are over 1.0 for all the blends in the entire low frequency range, which are also different from with those of reactive blends. Therefore, it can be inferred that the appearance of gelation-similar behavior is closely related with the compatibility and the phase structures of blends.

Figure 6 gives the curves of tanδ varied with φ of SEEPS at various ω for nylon1212/SEEPS blends. It can be observed that the different curves at different ω do not have a crossover similar to that of nylon1212/SEBS-g-MA blends, indicating that no gelation behavior exists in these blends. On the other hand, the curves shift downward along the tanδ axes with increasing ω, and the decreasing degree of tanδ is large when φ is less than 30 wt%. These changes indicate that there exists a structure transition for these blends.

Morphology analysis of blends

It is well known that the phase structures of the blends are mainly controlled by the components of the blends. When the concentration of the minor component increases, the particles begin to coalesce and form bigger structures. Further addition extends the continuous structure until the minor phase is continuous throughout the whole sample [47].

In order to explore further the relationship between gel point and morphology, we provide the SEM photographs of both blends. Figure 7 gives the SEM photographs of surface deal with the boiling xylene of nylon1212/SEBS-g-MA and nylon1212/SEEPS blends. For the nylon1212/SEEPS blends, SEEPS forms a dispersed phase and nylon1212 is a matrix. The phases of SEEPS distribute relatively orderly in the matrix of nylon1212, and the shape of SEEPS is almost spherical. With the increase in the concentration of SEEPS, the shape of dispersed phase has no change but the number and the size of disperse particles increase (Fig. 7a–c).

For the nylon1212/SEBS-g-MA blends, the sizes of minor phases are larger than those of nylon1212/SEEPS blends. When the concentration of elastomer is relatively low, the shape of elastomer phase is incompletely spherical and the sizes of elastomer phase are unequal (Fig. 7d). In the case of blends with 20 wt% SEBS-g-MA, the photograph shows that the nylon1212 phase forms a matrix and that the elastomer phase is very inhomogeneous (Fig. 7e). Some domains are very large with elongated shapes and other domains are very small with spherical shapes. The blends with 30 wt% SEBS-g-MA show obvious cocontinuous structure in some area (Fig. 7f). These morphology characteristics exhibit that blends undergo a change from dispersed droplets/matrix structure to cocontinuous morphology. In rheology, the viscoelastic behavior in the low frequency range can reflect the change of structure. The gel point we acquired is 17.45 wt% and should be

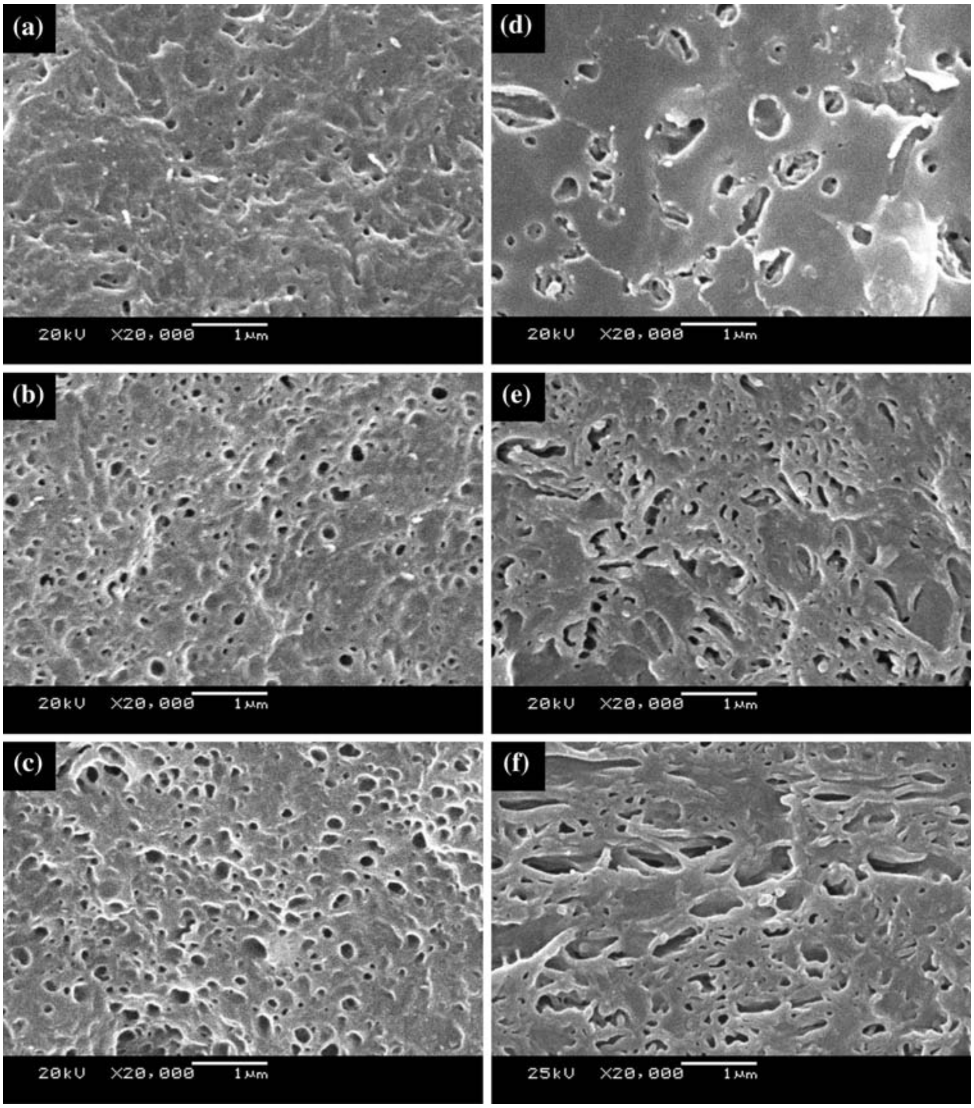


Fig. 7 SEM photographs of surface deal with the boiling xylene of nylon1212/SEBS-g-MA and nylon1212/SEEPS blends. (nylon1212/SEEPS blends, (**a**) 90/10, (**b**) 80/20, (**c**) 70/30; nylon1212/SEBS-g-MA blends, (**d**) 90/10, (**e**) 80/20, (**f**) 70/30)

corresponding to the concentration of structure change. Therefore, it can be concluded that the gel point for the reactive blends is a threshold of cocontinuous morphology, and morphology analysis can also be a method to determine the gel point.

It is difficult to conclude that what is a key symbol for emergence of gelation behavior in the blends. Based on above-mentioned results and researches for other blend systems, it can be inferred that such process can form in the blends that each component has strong interaction, such as chemical bond, hydrogen bond and other supermolecular interaction. These strong interactions can alter the macromolecular chain packing density and distribution, and phase structure, which is a dynamic process varying with component ratios of blends, temperature, and other factors. However, there are few studies focusing on this gelation process. It is still valuable for us to explore whether the emergence of gel point has some relationship with reaction degree for reactive blends, with hydrogen bond strength, and with phase structure.

Blending elastomer and plastics is an effective way to improve the impact strength of plastics. The impact strength usually has a maximum with the concentration of elastomer increasing, especially for the two phases reactive blends. The concentration of elastomer corresponding to the maximum can be called as the critical concentration. For the nylon and rubber blends, prior studies show that rubber particle concentration and size are important for improving the toughness of these blends [48, 49]. When the concentration of rubber attains a critical value, and the rubber particle size is in an appropriate range, the super-toughness nylon will be acquired. Wu combined these two interdependent parameters into one parameter, inter-particle distance, which acts as a critical parameter controlling toughness [50, 51]. Super toughness originates from the strong interaction between rubber particles, and matrix and rubber particle, while the form of gel depends on the change of macromolecular structures. Therefore, it is expected that the critical concentration of rubber is close to the gel point. If the critical concentration is close to the gel point, or has some deep correlation, a new way will be explored to determine the critical concentration, which will be discussed in future studies.

Conclusions

Studies on the gelation behaviors of the reactive blends of nylon1212 and functional elastomer were carried out. The reactive blends show an obvious transition from liquid-like behavior to solid-like behavior, indicating the gelation behaviors. The reasons are that the two components have an imidization reaction in the blending process and form graft copolymers, which can enlarge the length of macromolecule and improve the entanglement density. According to the method of Winter, the gel point (critical concentration of SEBS-g-MA) was calculated to be, $\varphi_g = 17.45$ wt% and the corresponding value of tanδ is 1.44. The gel index n calculated is 0.61 and the gel strength S_g is 1.08×10^4 Pa $s^{0.61}$. The non-reactive blends of nylon1212 and elastomer have no emergence of gelation behaviors. The gelation process can form in the blends that each component has strong interaction, such as chemical bond, hydrogen bond and other supermolecular interaction. The morphology analysis shows that the gel point for the reactive blends is a threshold of cocontinuous morphology and morphology analysis also can be a method to determine the gel point.

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