Progress in Organic Coatings 117 (2018) 146–153

Contents lists available at ScienceDirect

Progress in Organic Coatings

journal homepage: www.elsevier.com/locate/porgcoat

Effect of IR heating and the annealing time on the optical transmittance of polystyrene/Na-Alginate polymer composite films

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ARTICLE INFO

Keywords:
Composite films
Polystyrene
Sodium alginate
Optical transmittance
IR heating

ABSTRACT

Composite films of Na-Alginate (Alg) and Polystyrene (PS), which have different glass transition temperatures (T_g), with different weight ratios were prepared. Characterization of the film formation process was carried out by using the photon transmission (PT) measurements and the scanning electron microscopy (SEM) imaging. The composite films were annealed in the standard oven and under the IR lamp (IRH) for comparison. It was observed that IRH speeds up the film formation and decreases the required energy for film formation. On the other hand, the healing energies calculated for the films annealed in IRH were higher than those of the films annealed in the oven. In addition, it was shown that the optical transmittance of the films annealed in IRH increases as the annealing time increases while it is almost constant for the films annealed in the oven. Finally, it was observed that IRH causes some deformations on the surface of the composite films, especially at high temperatures, which may be due to the decomposition of low-T_g Alg polymers.

1. Introduction

Polymer latexes have been used in many areas of applications especially in the coating and adhesive technologies and biomedical field [1,2]. Polymer latex films are generally formed by coalescence of submicron polymer particles in the form of a colloidal dispersion, usually in water. The term “latex film” normally refers to a film formed from soft latex particles (glass transition temperature, T_g, below room temperature) where the forces accompanying the evaporation of water are sufficient to compress and deform the particles into transparent, void-free film. However, latex films can also be obtained by compression molding of a film of dried latex powder composed of relatively hard polymers, such as polystyrene (PS) or poly (methyl methacrylate) (PMMA) that has T_g above room temperature. [3,4]

Film formation from high and low T_g particles can occur in several stages. In both types, the first stage corresponds to the wet initial state. Evaporation of solvent leads to a second stage in which the particles form a close packed array. If the particles are soft, they are deformed to polyhedrons but hard latex, however, stay undeformed at this stage. Annealing of soft particles causes diffusion of polymer particles across particle-particle boundaries which lead the film to a homogeneous structure. Annealing of hard latex systems, deformation of particles first leads to void closure and then, after the voids disappear, diffusion across particle-particle boundaries starts [1,3,4].

Alginate polymers have a great potential in drug formulation due to their extensive application as food additives and their recognized lack of toxicity [5,6]. Ca-alginate was used to investigate release properties of Calcium Alginate Capsules Encapsulated Camphor Essential Oil Mixed with PS [7]. It was observed that as PS content increased, the limit release time was better than pure camphor only [7]. Also, alginate based composites in film structure are used in drug delivery [8] and this work will be important to understand the film formation process in detail for possible drug delivery system design. In another work, Na-Alginate was used with adding PS for the membrane separation where the pervaporation separation index (PSI) was considerably increased and excellent pervaporation performance was obtained [9].

IR heating (IRH) has some advantages comparing with the standard heating in the oven. One of them is the slow rate of heat transfer through air in convective heating which leads to long heat-up times. Using infrared emitters, it is possible to heat surfaces rapidly due to high transfer rate of energy and minimize the conduction losses. For example, drying time of aqueous solutions of poly vinyl alcohol (PVA) dropped from 120 min to 15 min for combined convective and IR radiative drying [10] which also shows that polymers absorb more energy effectively in the infrared range [11]. Another advantage is that the IR heating does not produce any combustion and does not need any

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https://doi.org/10.1016/j.porgcoat.2018.01.012
Received 12 May 2017; Received in revised form 20 October 2017; Accepted 9 January 2018
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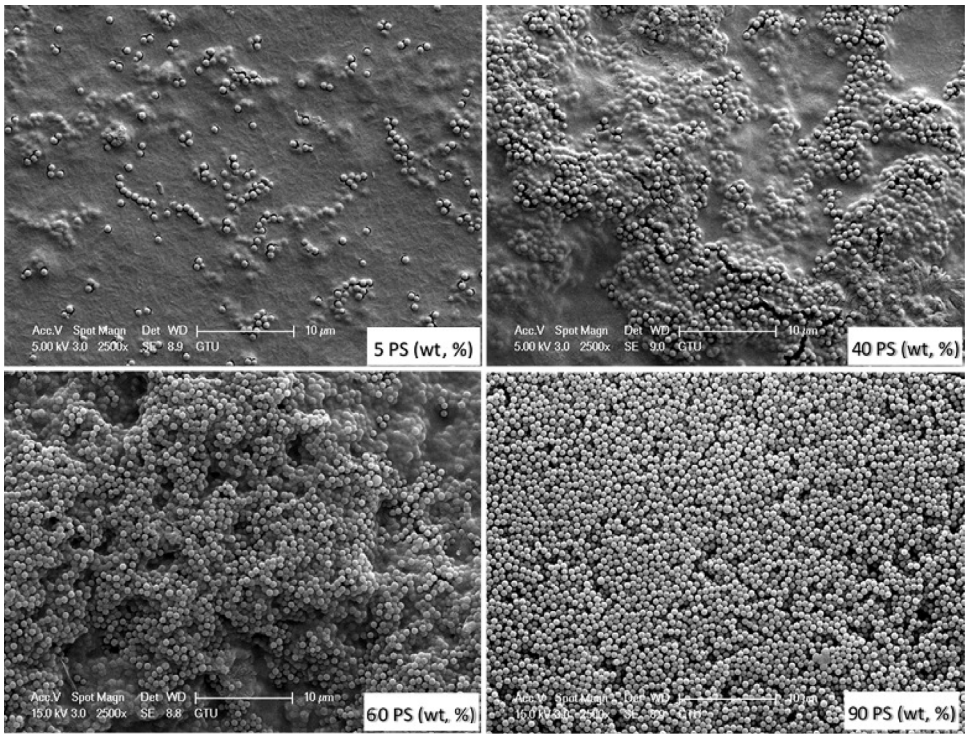


Fig. 1. SEM images of the films with different PS contents 5, 40, 60 and 90 (wt, %) and annealed at 60 °C in the oven.

recirculation of air, *i.e.*, it is clearer than the convectional heating. On the other hand, the combustion products, recirculation of dust, *etc.* make convective heating unsuitable for "clean" applications. Third, the time to reach the operating temperature for the convection ovens, especially for high temperatures, lasts from 30 min to 2 h depending on the size of the convection oven, which means that the oven must be turned on much before the experiment to reach the required temperature. However, for infrared heating this time is very short because it can be switched on and off within very short time intervals and by adjusting the electrical voltage applied on the emitter it can reach the operating temperature in a very short time. Fourth, in standard heating in an oven, heat diffuses through the film surface from the substrate and the time for homogeneous distribution of temperature through the film directly depends on this heat diffusion. Heat diffusion and evaporation rate of water in waterborne coatings for the film formation is faster when IRH is used instead of standard heating in the oven. Fifth, the emitted radiation from IR source is only absorbed by the film (or treated portions of the film) and not by the surrounding air or objects [12].

Different techniques were used to characterize the latex film formation such as, small-angle neutron scattering (SANS) method at molecular level by Sperling and co-workers on compression-molded polystyrene (PS) film [13]. Direct-nonradiative energy transfer (DET) method was employed to investigate the film formation process from dye-labeled polymeric particle [14–17]. Steady state fluorescence technique combined with DET was used to examine healing and interdiffusion processes in the dye labeled poly (methyl methacrylate) (PMMA) latex systems [18]. Photon transmission method has been performed to study latex film formation from PMMA and PS particles, using the UV–vis (UVV) technique as a function of temperature and time [19].

In the present work, we aimed to monitor the film formation mechanism from high-T_g PS and low-T_g Alg composites varying 5–90 PS (wt, %) using UV–vis (UVV) photon transmission technique with two selective heating systems, IRH and oven at three different annealing times. During the film formation process, the transmittance at 800 nm was observed to carry out the film formation process and the effect of the annealing systems together with the annealing times. Prager and Tirell (PT) [20] method was employed to investigate the healing processes at the junction surfaces.

2. Experimental

PS particles was produced *via* surfactant free emulsion polymerization process. The polymerization was performed batch-wisely using a thermostated reactor equipped with a condenser, thermocouple, mechanical stirring paddle and nitrogen inlet. The agitation rate was 400 rpm and the polymerization temperature was controlled at 90 °C. Water (100 ml) and styrene (5 g) were first mixed in polymerization reactor where the temperature was kept constant (at 90 °C). Potassium peroxodisulfate (KPS) initiator (0.1 g) dissolved in small amount of water (2 ml) was then introduced in order to induce styrene polymerization. The polymerization was conducted during 18 h. PS particles prepared in this way have an average size of 600 nm and a glass transition temperature of 90 °C.

Alginic acid sodium salt in powder form was obtained from Sigma-Aldrich and used as received. The glass transition temperature of Alg was measured as 29 °C by using differential scanning calorimetry.

100 ml 2.4% PS and 100 ml 1% Alg stock solutions were prepared. Two sets of films were prepared from the dispersion of PS/Alg composites by placing the same number of drops on a glass plates with the size of 1.4 × 2.5 cm^2 and allowing the water to evaporate. One set was prepared for annealing in the convective heating in the oven and the other set was prepared for annealing in IRH. In each set, the weight ratio, ratio of the weight of PS to the weight of Alg, of the composites was changed from 5/95 to 90/10. Then the samples were separately annealed starting from 40 °C below T_g of PS to 270 °C for 10 min in both oven and in IRH. The temperature was maintained within ± 2 °C during annealing for oven and ± 1 °C for IRH.

Photon transmission measurements were observed by using UV–vis spectrometer, B&W Tek. I_{tr} of the films were detected at 800 nm. A glass plate was used as substrate for all transmission experiments and measurements of the transmittance of the annealed composite films were carried out at room temperature. Scanning Electron Microscopy (SEM) images of pure PS and some of the PS/Alg composite films were obtained by Philips XL30 SFEG probe microscopy.

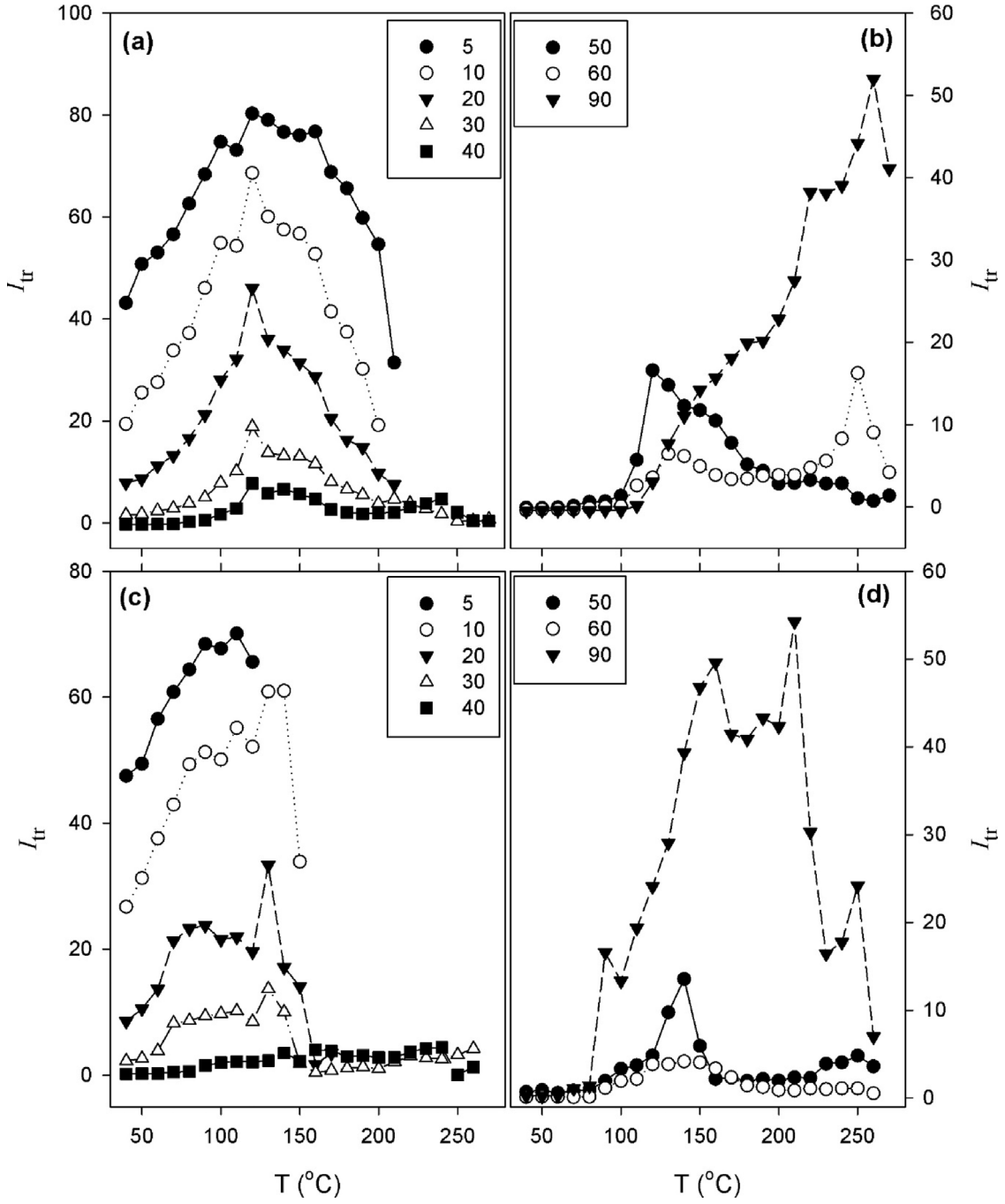


Fig. 2. The variation of the transmitted light intensity at 800 nm depending on the annealing temperature for the composite films annealed in the oven (a, b) and in IRH system (c, d). The numbers in the legends show the relative PS weight content in the films.

3. Results and discussion

The SEM images of the films with different PS contents 5, 40, 60 and 90 (wt, %) annealed at 60 °C in the oven were given in Fig. 1. PS particles, which are seen as white spherical polymer particles in the SEM images, can preserve their structures at 60 °C due to their high glass transition temperature. On the other hand, since Alg polymer particles have lower glass transition temperature they cannot preserve their structures and flow through each other at 60 °C. In the SEM images, they are seen as continuous matrix, dark grey regions in the images. Another point in these images that the PS particles tends to come close each other in the composite film to form phase separated domains and thus the film structure is made of individual coalesced PS domains immersed in a continuous matrix of Alg.

The temperature dependence of the transmitted light intensity at 800 nm for PS/Alg composite films with different compositions were represented in Fig. 2. Standard film formation behaviors were observed for these composite films as we observed in our previous works [21,22], *i.e.*, the intensity of the transmitted light starts to increase after a certain onset temperature, called the minimum film formation temperature T_o, and then reaches a maximum with increasing annealing temperature. Finally, it decreases above another important temperature that is called as healing temperature, T_h.

The following results can be drawn from Fig. 2: (*i*) especially, for low PS content (below 60%, wt), an overall decrease in the transmitted light intensity was observed as the PS content is increased. This is due to the scattering behavior of the voids in the film. PS particles tends to come close each other in the composite film to form phase separated

domains which is dominant especially at low PS contents as seen in Fig. 1. Film structure is made of individual coalesced PS domains immersed in a continuous matrix of Alg. Up to some weight content of PS, which depends on the properties of the other components in the composite film, these voids increases the probability of the scattering of the light passing through the composite film [23,24]. (*ii*) For high PS content (especially above 60%, wt), an opposite of the behavior in (*i*) was observed. The intensity of the transmitted light increases as the PS content is increased (Fig. 2(b) and (d)). This is an expected result, because when the PS content is increased it becomes dominant in the film and the PS particles could change the morphology of the films to more homogeneous structure by flowing through the voids. However, the film formation temperature of the composite films with high PS content is higher than the films with lower PS content due to the high T_g of PS particles. (*iii*) When the results of the annealing systems are compared, Fig. 2(a)–(c) and Fig. 2(b)–(d), it is clearly seen that the film formation process is completed at lower temperatures in IRH system. This result is in agreement with our previous results given in reference [21] which shows that the heat is transported to the film more effectively in IRH system.

The SEM images of the films with PS content 90 (wt, %) before annealing and after annealing at 150 °C in the oven and in IRH were given in Fig. 3. As seen from Fig. 3(a) that the film structure is dominated by the PS particles. When the composite films were annealed, the PS particles flowed through the voids and the size of the voids decreased as seen in Fig. 3(b) and (c). However, some of the PS particles in the film annealed in the oven still preserve their structures while the PS particles were completely melted when they were annealed in IRH even if the annealing temperatures are equal for each annealing system. These SEM images clearly shows the efficiency of IRH system for annealing the composite films. In addition, some large pores/voids occurred on the surface of the films as a result of annealing in IRH system at high temperatures as seen in Fig. 3(c). These large pores are probably due to the decomposition of some of the Alg polymers on the surface of the film.

The variations of I_{tr} with respect to PS content were represented in Fig. 4. In Fig. 4(a), I_{tr} is decreasing with both temperature and PS content from 5 to 60 (wt, %) for the films annealed in the oven. This result is understandable because at this temperature interval, a phase separation between PS and Alg occurs and PS particles construct new clusters due to the aggregation. As a result of this relocating of the particles to construct clusters, some big voids are occurred between the clusters and I_{tr} decreases due to the increasing probability of the scattering. But for the PS contents above 60 (wt, %), the intensity increases with the increasing PS content. Because at these contents of PS, the dominant component of the composite film is PS and it spreads through the film homogeneously. Also, for these PS contents the transmittance increases with the increasing temperature as expected (Fig. 4(b)). For the films annealed in IRH system, especially for low temperatures similar behaviors were observed with the films annealed in the oven.

T_o and T_h values were presented in Fig. 5. T_o is often used to show the minimum possible temperature for the polymer particle deformation which is sufficient to decrease interstitial void diameters to sizes well below the wavelength of light [25]. Below this critical temperature, the dry latex is opaque and powdery. However, at and/or above this temperature, a latex film becomes continuous and clear film is obtained [26]. Therefore, T_o has been considered in this study as the temperature above which the I_{tr} starts to increase. T_h is used to define the temperature at which the chains almost start to gain sufficient kinetic energy to transform their center of masses across the junction surface (interdiffusion) [20,21].

As seen from Fig. 5(a) that T_o is increasing up to a certain temperature with increasing PS content and then decreases for both heating systems used for annealing. This observation points out that T_o is strongly influenced by the amount of high-T_g PS. For very low values of PS content, 5 and 10 (wt, %), the onset temperature is constant and above this PS content, it increases as the PS content increases. Up to 50 (wt, %) PS content, no difference was observed between the heating systems used for annealing the composite films. These two intervals can be explained by the low-T_g value of Alg in the composite films. At low PS contents Alg is dominant in the system and since Alg has smaller glass transition temperature it can move easily at lower temperatures. This property makes the composite films insensitive to the types of the heating system and no difference was observed as seen in Fig. 5(a). For the films with higher PS content, above 50 (wt, %), the onset temperatures observed for IRH system are slightly smaller than those observed in the oven. This clearly indicates that the annealing the films in IRH decreases the required temperature to start the film formation process as mentioned before and it is in good agreement with the results of our previous work [21]. Another point in Fig. 5(a) is that, T_o values increase up to 80 PS (wt, %) content and then decrease for both systems. This situation is related to a phase separation which is more effective in the low PS content as seen in Fig. 1, *i.e.*, the PS clusters constructed as phase separation behave like scattering centers at low concentration and the onset temperature increases as a result of increasing PS concentration until a specific concentration. Above this specific concentration, PS particles become dominant and the voids can be closed easier by the huge amount of PS particles, which results in a decreasing onset temperature.

The dependence of the healing temperatures, T_h, on the PS content was given in Fig. 5(b). Here, a similar behavior with the behavior of the onset temperature was observed. Again, at low PS contents, below 50 (wt, %), the healing temperatures are close to each other for each heating system but at high PS contents the healing temperatures for the films annealed in IRH are smaller than those observed for the films annealed in the oven. This again indicates that the annealing the films in the IRH system decreases the required temperature for the interdiffusion stage of the film formation process.

This result may show that the thermal energy emitted by the IR lamp is absorbed more efficiently by the high amount of PS particles. At

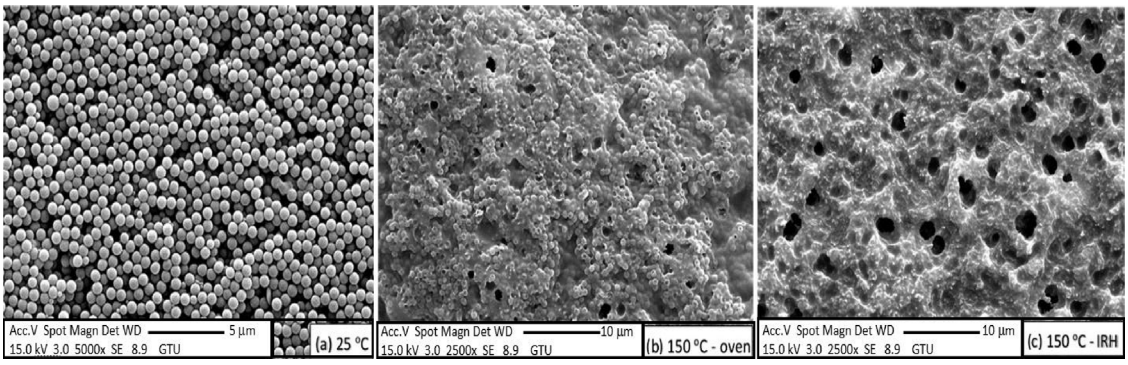


Fig. 3. SEM images of the films with PS content 80 (wt, %) before annealing (a) and after annealing at 150 °C in the oven (b) and in IRH (c).

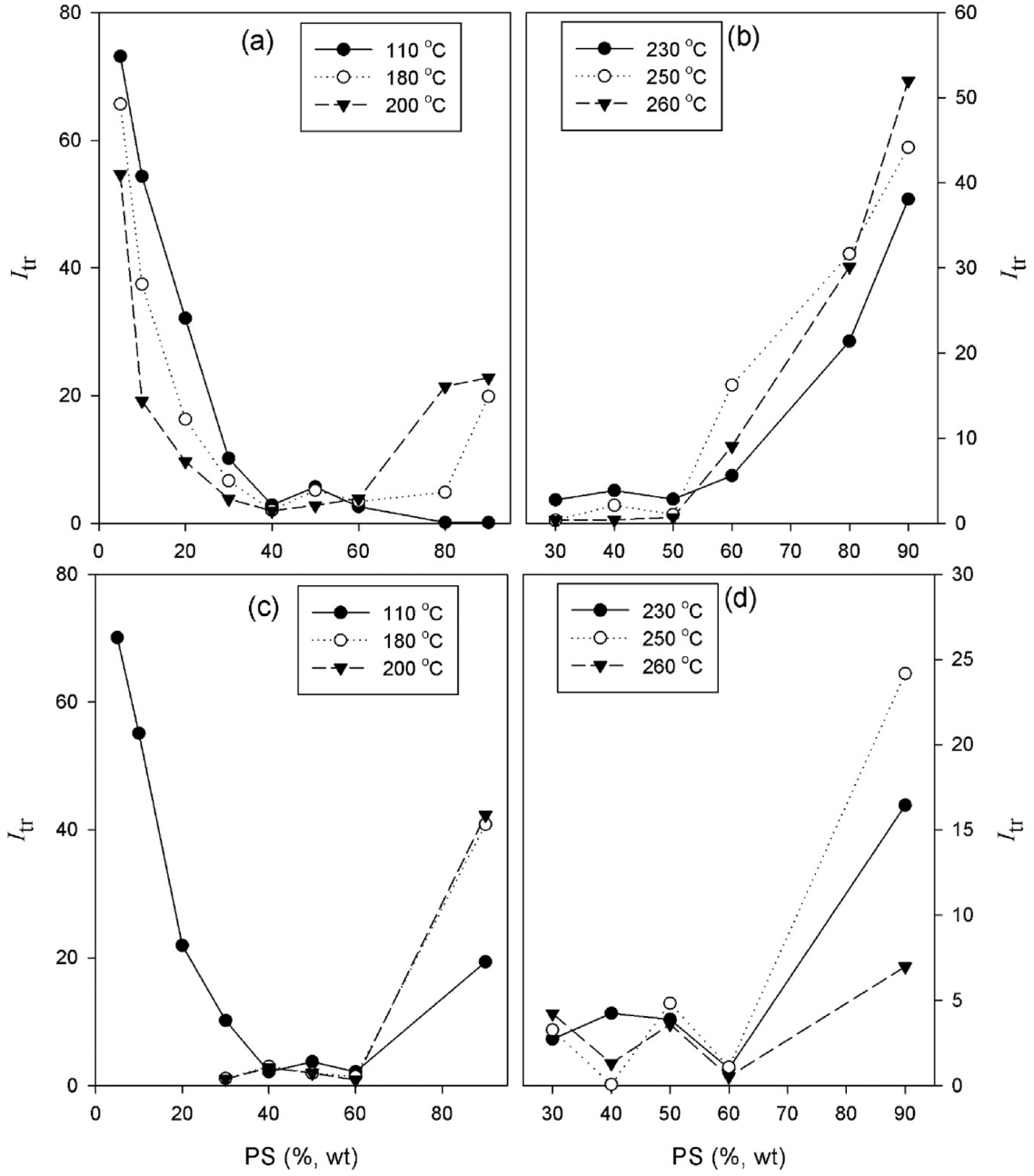


Fig. 4. The variation of the transmitted light intensity at 800 nm depending on the PS content for the composite films annealed in the oven (a, b) and in IRH system (c, d) for 10 min. The numbers in the legends show the relative annealing temperatures.

high PS concentrations, since the thermal energy is absorbed more efficiently by the PS particles, the healing and void closure process are achieved at lower temperatures by IRH.

3.1. Calculation of the activation energies

The activation energies for the void closure and the interdiffusion processes are calculated by using the following equations [19]

$$I_{tr}(T) = S(t)\exp\left(-\frac{3\Delta H}{kT}\right) \quad (1)$$

$$I_{tr}(T)/I_{tr}(\infty) = A\exp\left(-\frac{\Delta E}{2k_B T}\right) \quad (2)$$

where $S(t) = (\gamma t/2AC)^3$, k_B is the Boltzmann's constant, ΔH and ΔE are the activation energies of the void closure and the interdiffusion stages, respectively, and I_{tr} is the transmittance at 800 nm. The parameters γ, t, and C in $S(t)$ are the surface energy, time, and integration constant, respectively. The parameter A is defined as $A = (N_A h/V)e^{-\Delta S/kB}$ where N_A is Avagadro's number, h is Planck's constant, V is the molar volume, and ΔS is the entropy change. Detailed descriptions and derivations of the Eqs. (1)–(2) and the parameters was given in Reference [19].

Calculated activation energies for the PS/Alg composite films annealed by the convective heating in the oven and IRH were given in Fig. 6. As it was already discussed earlier that the increase in I_{tr} originates because of the void closure process and Eq. (1) was used between T_o and T_h for all composite films annealed in both oven and IRH systems. The calculated activation energies for the void closure process, ΔH, were given in Fig. 6(a) for two heating systems. Required force for the film formation depends on the inverse of the particle size according to the descriptions of the film formation driven by capillary forces [27]. Since Alg has lower T_g than PS particles, ΔH values are smaller for the

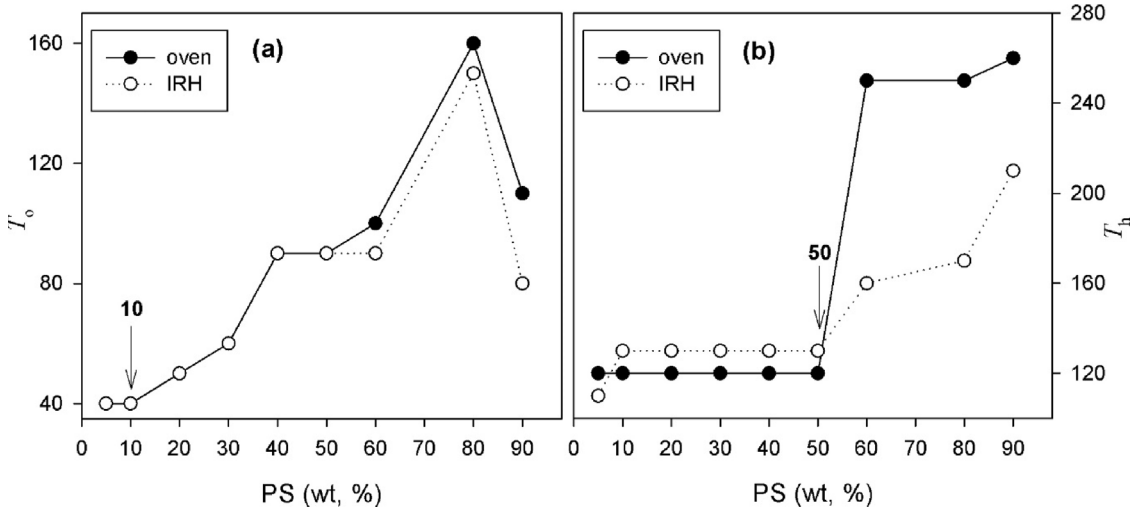


Fig. 5. Variation of the onset, T_o, and the healing, T_h, temperatures with respect to the PS content for the composite films annealed in the oven (filled circle) and in IRH system (open circle).

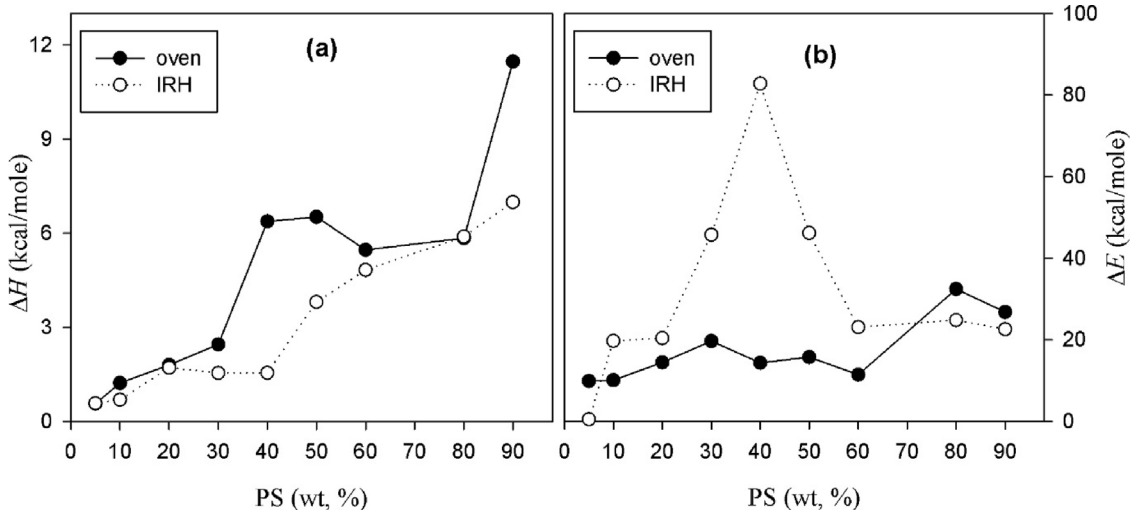


Fig. 6. Variation of the void closure (a), and the interdiffusion (b) activation energies with respect to the PS content for the composite films annealed in the oven (filled circle) and in IRH system (open circle).

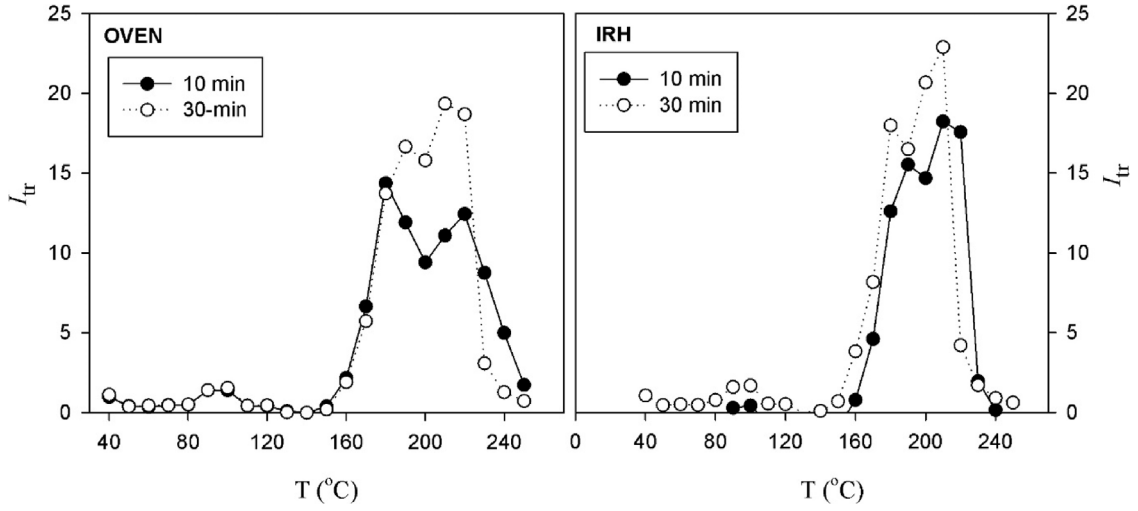


Fig. 7. Transmittance at 800 nm versus temperature for different annealing times for the films annealed in the oven (a) and in IRH (b). The numbers given in the legends show the annealing time.

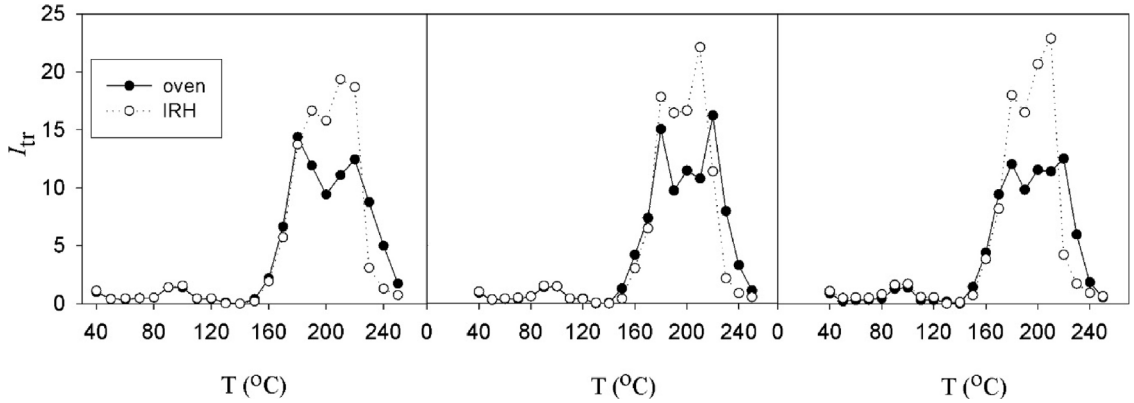


Fig. 8. Transmittance at 800 nm versus temperature for the films annealed in the oven (filled circle) and in IRH (open circle) at different annealing times; 10, 20, and 30 min.

films with low PS content annealed in both IRH and oven. When the PS content is increased ΔH also increases due to the higher glass transition temperature of PS. In addition, ΔH values calculated for the films annealed in IRH are smaller than those calculated for the films annealed in the oven. This shows that the thermal energy is transported more effectively in IRH system and this decrease the required energy to move the particles through the voids.

The calculated activation energies for the interdiffusion process, ΔE, were given in Fig. 6(b). When PS content is increased in the composite film, ΔE also increases for both heating systems. This point is related to the high glass transition temperature of PS particles and when the concentration of PS particles is increased in the film, the required energy to move the polymer chains through the junction will increase. At moderate levels of PS content (around 40%, wt), the activation energy for interdiffusion process observed in IRH are higher than those observed in the oven. This shows that at these PS contents the oven is more effective than the IRH system. This point also was observed in our previous work on PS/PAAm composite films [28].

In addition, the activation energies of the interdiffusion process are much larger than the activation energies of the void closure process for both annealing systems. This is expected result, because a single chain needs more energy to overcome the diffusion across the polymer–polymer interface than to be accomplished by the viscous flow process.

In Fig. 7, the variations of the transmittance at 800 nm with respect to temperature were given for the films with PS content 80 (wt, %) annealed in the oven and in IRH. Almost no difference was observed between the shorter and longer annealing times for the films annealed in the oven. However, for the films annealed in IRH, some increase was observed when the film was annealed for a longer time. This result may indicate that the conformational degrees of freedom of the film structure for the films annealed in the oven is less than the films annealed in IRH. Thus, the film structure obtained as a result of 10 min annealing in the oven has a quasi-stable structure and increasing the annealing time will not affect the structure considerably. On the other hand, the film structure reached as a result of annealing in IRH system probably has some more homogeneous conformations and increasing the annealing time, it can be possible to convert the film structure to more homogeneous nature.

Fig. 8 shows the change of I_{tr} with respect to the annealing temperature for the films with PS content 80 (wt, %) depending on the annealing times (10, 20 and 30 min) in the oven and in IRH. It is clearly seen that for all annealing times the transmittance is bigger for the films annealed in IRH and the difference between the oven and IRH increases as the annealing time is increased. In other words; when the films are annealed at longer time, films become more homogenous and decreases the radius of the voids which results in higher transparency in IRH.

4. Conclusions

The following points can be concluded from the experiments performed in this work.

i The film formation process is completed in lower tempertures when IRH system is used for annealing. This clearly indicates that IRH is more effective than the standard heating in the oven.
ii The required energy to start the film formation also decreases in IRH system especially for films include high-T_g polymer particles. This shows that the thermal energy is transported to the polymer particles in the composite film more effectively in IRH system.
iii If the low- T_g polymer particles are dominant in the composite film, the type of the heating system used for annealing does not affect the film formation process.
iv When the annealing time was increased from 10 min to 30 min, no considerable change in the optical transmittance of the films annealed in the oven was observed. On the other hand, the optical transmittance of the films annealed in IRH was increased as the annealing time was increased.

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