

Fabrication and Apparent Kinetic Study of Poly(methyl methacrylate)/Poly(octyl acrylate) and Poly(octyl acrylate)/Poly(methyl methacrylate) Latex Interpenetrating Polymer Networks

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ABSTRACT: Two latex interpenetrating polymer networks (LIPNs) were synthesized with methyl methacrylate (MMA) and octyl acrylate (OA) as monomers, respectively. The apparent kinetics of polymerization for the LIPNs was studied. This demonstrates that network II does not have a nucleus formation stage. The monomers of network II were diffused into the latex particles of network I and then formed network II by *in situ* polymerization. It indicates that the polymerization of network I obeys the classical kinetic rules of emulsion polymerization. But the polymerization of network II only appears a constant-rate stage and a decreasing-rate stage. The apparent activation energies (E_a) of network I and network II of PMMA/POA

were calculated according to the Arrhenius equation. The E_a values of POA as network I (62 kJ/mol) is similar to that of POA as network II PMMA/POA (60 kJ/mol). However, the E_a value of PMMA as network II POA/PMMA (105 kJ/mol) is higher than that of PMMA as network I (61 kJ/mol). Results show that the E_a value of the network II polymerization is related to the properties of its seed latex. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 116: 2126–2132, 2010

Key words: latex interpenetrating polymer network; methyl methacrylate; octyl acrylate; kinetic; apparent activation energy

INTRODUCTION

Emulsion polymer latexes are used in a wide variety of applications, such as synthetic rubber, paints, adhesives, leather treatment, impact modifiers for plastic matrices, additives for construction materials, and flocculants due to their benign environmental and safe characteristics. They are also used in biomedical and pharmaceutical applications such as diagnostic tests and drug delivery systems.^{1,2} Among the polymer latexes, the composite latex particle is an extremely important class of material known to exhibit a variety of unique properties. Interpenetrating polymerization is a mode of blending two or more polymers to produce a mixture, in which phase separation is not as extensive as in nor-

mal blending or mixing. The latex interpenetrating polymer network (LIPN) is a special category of composite latex particles and has gained importance in 1990th.³ LIPNs have attributes of involving relatively simple synthetic routes with the potential to combine the advantages of thermosets with those of thermoplastics.^{4,5} Thus LIPNs have received much attention in both scientific and industrial studies because they can often greatly improve mechanical or other properties of polymers through the unique topological entanglement and interpenetration between networks.^{6–8} They have prodigious application potentials in plastic modification, rubbers, coatings, damping materials, medical materials, etc.^{9–12}

LIPNs are generally synthesized by a two-stage emulsion polymerization technique. The first monomer is emulsion-polymerized and cross-linked to form a seed latex (polymer network I). Monomer II with a crosslinking agent is added into the seed latex and then polymerized *in situ* without forming fresh particles.^{13–18} A variety of complex factors, such as the method of monomer addition, the monomer ratio and the hydrophilicity of monomers, is involved in the synthetic process, the ultimate morphology of the obtained LIPNs and the mechanical properties of the films.^{19–23} Studies on LIPNs have mainly focused on the preparation process,

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morphology, and properties.^{24–29} Very little information has been reported on the kinetic features, and in particular, the apparent kinetics of the polymerization process.

Regarding the kinetics of traditional emulsion polymerization, the well known Smith-Ewart model is mostly applicable for the course of polymerization.^{30,31} The emulsion polymerization process usually consists of three intervals. In Interval 1, i.e., the nucleation stage, of classical emulsion polymerization, the rate of polymerization increases with the progress of polymerization as a result of increased numbers of polymer particles until particle nucleation ceases, typically at 10% conversion. In Interval 2, the particles grow steadily until all monomer droplets disappear. In this interval, the total polymer particle number and the monomer concentration in the particles are usually considered to be constant. The rate of polymerization is relatively constant. In Interval 3, the monomer concentration decreases with increasing conversion and polymerization proceeds under the monomer starved condition, so that the rate of polymerization decreases.

The emulsion and seed emulsion polymerization of acrylate monomers has been studied by many authors.^{32–35} In 1990, Tobita et al. investigated the crosslinking kinetic model for bulk polymerization and emulsion polymerization.¹⁸ They pointed out that the difference between bulk polymerization and emulsion polymerization was that the reaction rate of the former was slower than that of the latter. The reason was because the viscosity of the bulk polymerization was high and the efficiency of the initiator was low. Zhang³⁶ studied the kinetics of the polymerization process and the influence of the initial concentration of the emulsifying agent, initiator and the extent of crosslinking. Zou et al.^{37,38} and Sahoo³⁹ investigated the synthesis of crosslinking latex. They found that different monomers possessed different kinetic models, but the common characteristic was that the reaction rate increased with the increment of crosslinking density. As to the polymerization of LIPN emulsion, its polymerization is carried out using network I as the seed emulsion. Consequently, the kinetics of polymerization differs from traditional emulsion polymerization. The results of polymerization kinetics can be used to predict the morphology of the latex particles, formation of the particles (nucleation) and the particle size distribution.^{40,41} This ability to control particle morphology would significantly enhance the physical properties of these materials and their application.

We previously studied the apparent kinetics of polymerization of LIPNs of monomers styrene (S) and ethyl acrylate (EA).⁴² In this work, using monomers methyl methacrylate (MMA) and octyl acrylate (OA), two latex IPNs (PMMA/POA LIPN and

POA/PMMA LIPN) were synthesized with divinyl benzene as crosslinking agent. This study examined the mechanism of the apparent kinetics of the polymerization process for these latex IPNs. To gain a better understanding of the apparent kinetics for different LIPNs, the apparent activation energies of the polymerization process for LIPNs with different seed latexes were calculated. An attempt has been made to investigate the relationship of apparent activation energy of different LIPNs.

EXPERIMENTAL

Materials

MMA and OA were purchased from Tianwei Chemical Co. (Tianjin, China) and distilled under vacuum before use. Divinyl benzene (DVB) was washed with aqueous NaOH to remove inhibitors, then dried for several hours with MgSO₄ and distilled under reduced pressure. Potassium persulfate (KPS, analytically pure), sodium bicarbonate (NaHCO₃, analytically pure), polyoxyethylene nonylphenol (OP-10) were obtained from Beijing Chemical Plant (Beijing, China) and used without further purification.

Polymerization procedure

Polymer network I (the seed latex) polymerization

Polymerization was conducted in a 500 mL round bottomed, four-neck flask fitted with a mechanical stirrer, nitrogen inlet, condenser, and pipette inlet. The reactor was immersed in a thermostatted water bath to maintain constant temperature. The typical recipe for PMMA/POA LIPN is given in Table I. The recipe for other LIPNs is the same as that of PMMA/POA LIPN, only different monomers are used in network I and network II. Deionized water and emulsifier were stirred and gently warmed in the flask. Monomer I mixture were added to the system under constant stirring speed (150/rpm) at the specified temperature. Then initiator was added into the reaction system. At the same time, the time to carry out the polymerization of network I was noted.

Polymer network II (LIPN) polymerization

After the preceding polymerization had been performed, a mixture of monomer II was added to the seed latex (network I). Then the initiator was added and the time noted simultaneously. The polymerization reaction was carried out under constant temperature. Except for no added emulsifier, the polymerization process of network II was the same as that of the seed latex.

TABLE I
Typical Recipe for the Synthesis of LIPN PMMA/POA

Component	Network I (seed latex)	Network II (LIPN)
MMA (g)	19.6	
OA (g)	–	29.4
DVB (g)	0.4	0.6
OP-10 (g)	1.5	–
NaHCO ₃ (g)	0.1	0.15
KPS (g)	0.1	0.15
Deionized water (mL)	130	10

Characterization

Calculation of polymerization conversion

The monomer conversion, the percent of polymerized monomers to all monomers added into system, was determined by a conventional gravimetric method. At a certain time during the seed latex polymerization or the network II polymerization, a sample of the emulsion latex was taken out of the reactor and poured into methanol with hydroquinone to stop the reaction. The precipitated polymers were washed with methanol and water several times, and then dried in a vacuum oven.

Calculation of the rate of polymerization and the apparent activation energy of the polymerization process

The conversion was calculated by the aforementioned method at a certain interval. The curve of conversion versus time was obtained at different temperatures. The rate of polymerization could be evaluated by differential calculation of the curve of conversion versus time (C%-t). Then the curve of polymerization rate versus conversion (R_p -C%) could be described. The variation of rate was presented by the R_p -C% curve on the process of polymerization. As with the classical emulsion polymerization, the process included an increasing-rate interval, a constant-rate interval and a decreasing-rate interval according to the well known Smith-Ewart theory.³¹ The majority of the monomer had been reacted to polymer in the constant-rate interval. The apparent activation energy in the constant-rate interval was calculated according to the Arrhenius equation.^{31,41}

$$R_p = A \cdot e^{-E_a/RT} \quad (1)$$

$$\ln R_p = -E_a/RT + \ln A \quad (2)$$

A line was obtained from the relationship of $\ln R_p$ to $1/T$. E_a was calculated from the gradient K of the line.

$$E_a = -KR \quad (3)$$

Where R_p is the rate of polymerization; A , a constant; R , the gas constant; T , the polymerization temperature; E_a , the apparent activation energy; K , the gradient of the line.

The particle size and size distribution of latexes were determined by dynamic light scattering (DLS) using a 90 Plus particle size analyzer (Brookhaven Instrument Corporation). The samples of latexes were diluted with distilled water to adjust the solid content to around 0.05 wt % and were placed directly in the cell. All measurements were carried out at 25°C at a fixed angle of 90°, and the measurement time was 3 min.

RESULTS AND DISCUSSION

Effect of concentration of OP-10 on MMA/OA polymerization

The emulsion polymerization of MMA (network I) was conducted with various concentrations of OP-10 at a constant temperature of 75°C. The curve of conversion versus time is shown in Figure 1(a). It can be seen that conversion increases almost linearly with time over the conversion range from 10 to 40%. After higher conversion around 70%, the rate of polymerization R_p increases slightly. It may be partly attributed to the gel effect.³¹ But the lower concentration of monomer and free radicals makes R_p increase slightly till to the constant-rate stage. In addition, the rate of polymerization increases with increasing concentration of OP-10. The propagation reaction of free radicals with monomer occurs in the monomer-swollen micelle. A high concentration of surfactant produces a high number of micelles, which leads to a high rate of polymerization. The emulsion polymerization of PMMA/POA LIPN was carried out with the earlier obtained PMMA latex as seed emulsion. Figure 1(b) shows the curves of conversion versus time. The polymerization rate of network II PMMA/POA also increases with the increase in concentration of OP-10 in the seed latex. During the polymerization of network II, no surfactant was supplied to the system, so no new nucleation occurred. The polymerization of network II (POA) occurred in the seed latex (network I PMMA). Consequently, a high concentration of surfactant results in more particles of seed latex, which leads to a high polymerization rate of network II (POA).

Effect of crosslinking agent concentration on MMA/OA polymerization

The temperature of the two network polymerizations was held at a constant 75°C and the other variables such as monomer and emulsifier concentration

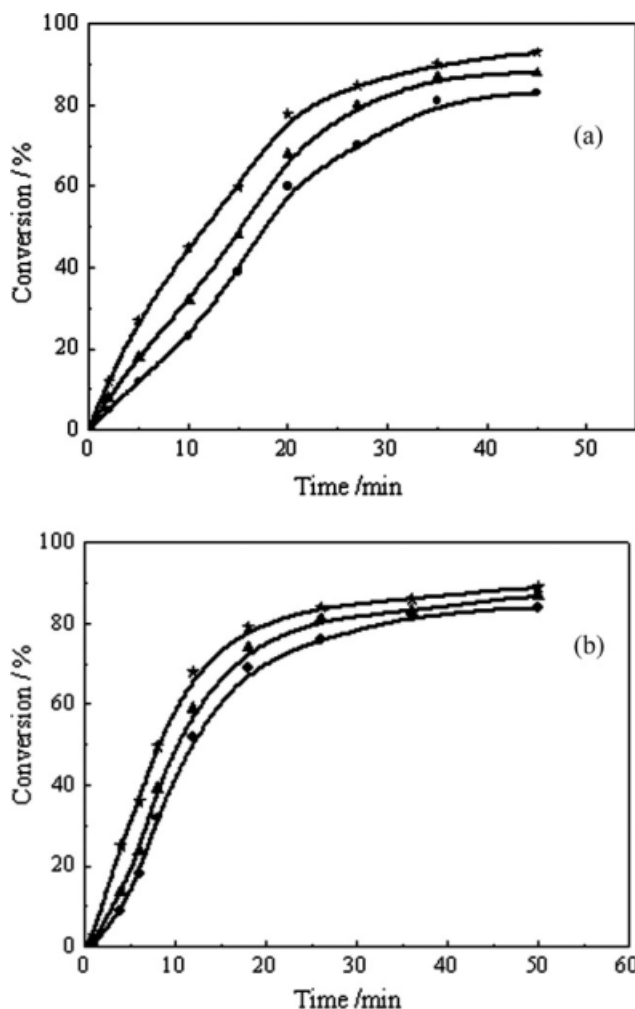


Figure 1 (a) The curves of conversion versus time of PMMA at different concentration of OP-10 (75°C, DVB 2%). ●, 0.020M; ▲, 0.025M; ★, 0.030M. (b) The curves of conversion versus time of PMMA/POA LIPN at different seed emulsion synthesized with different concentration of OP-10 (75°C, DVB 2%). ●, 0.020M; ▲, 0.025 M; ★, 0.030 M.

remained unchanged in the experiments. The concentration of crosslinking agent DVB was varied from 1 to 3% (molar ratio of DVB to monomers). Figure 2(a) shows a plot of conversion versus time of PMMA (network I). This indicates that conversion increased with the increase in DVB. Figure 2(b) shows a plot of conversion versus time of PMMA/POA (network II) synthesized by varying DVB from 1 to 3%. The polymerization rate was found to be highly sensitive to changes in crosslinking agent DVB irrespective of the polymerization of network I and network II. It is possible that the crosslinking action interferes with the movement of molecular chain. Consequently, the restricted termination of growing radicals within the monomer-polymer particles gives rise to very rapid polymerization and the formation of higher molecular weight polymers.³¹

Polymerization kinetics of LIPN network I (seed latex PMMA) at different temperatures

Figure 3(a) shows the evolution of instantaneous and overall conversion versus time for emulsion polymerization of network I (PMMA), carried out at different polymerization temperatures. It can be seen from the curves that there is an initial period of very low instantaneous conversion, especially at lower temperature. This slow start to polymerization is due to the low nucleation. Under these initial conditions, monomers and radicals resulting from the decomposition of initiator need to diffuse into the micelle to form latex particles for nucleation.³¹ Then the propagation reaction progresses from the primary latex particles. The rate of polymerization could be evaluated to obtain Figure 3(b) by differential calculation from the curve in Figure 3(a). This shows that the synthesis of network I, the production of seed latexes, obeys the classical kinetic rules

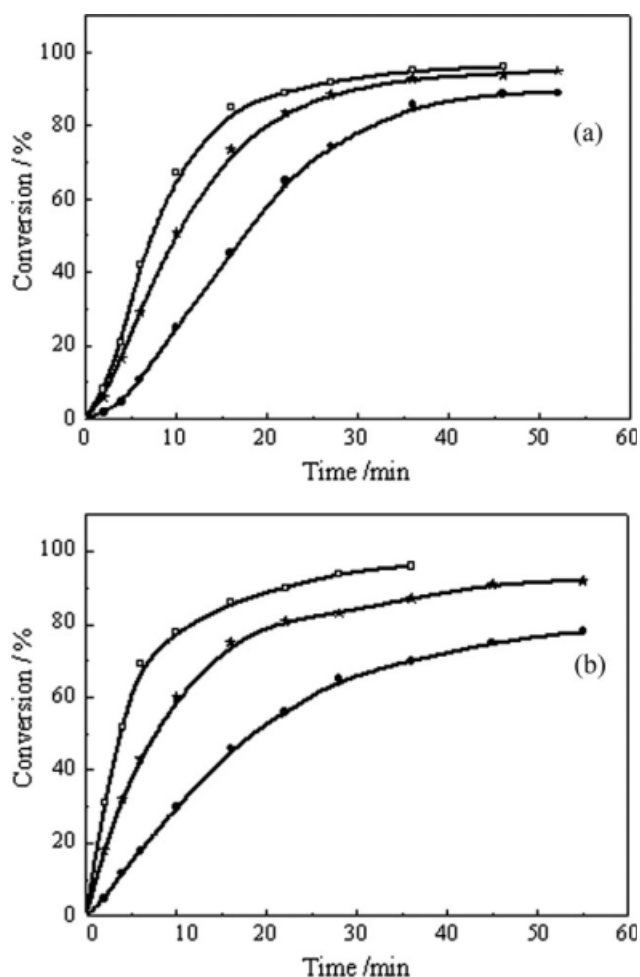


Figure 2 (a) The curves of conversion versus time of PMMA (network I) at different content of crosslinking agent DVB (75°C, OP-10 0.025 M). ●, 1%; ★, 2%; □, 3%. (b) The curves of conversion versus time of PMMA/POA LIPN (network II) at different content of crosslinking agent DVB (75°C, OP-10 (0.025 M). ●, 1%; ★, 2%; □, 3%.

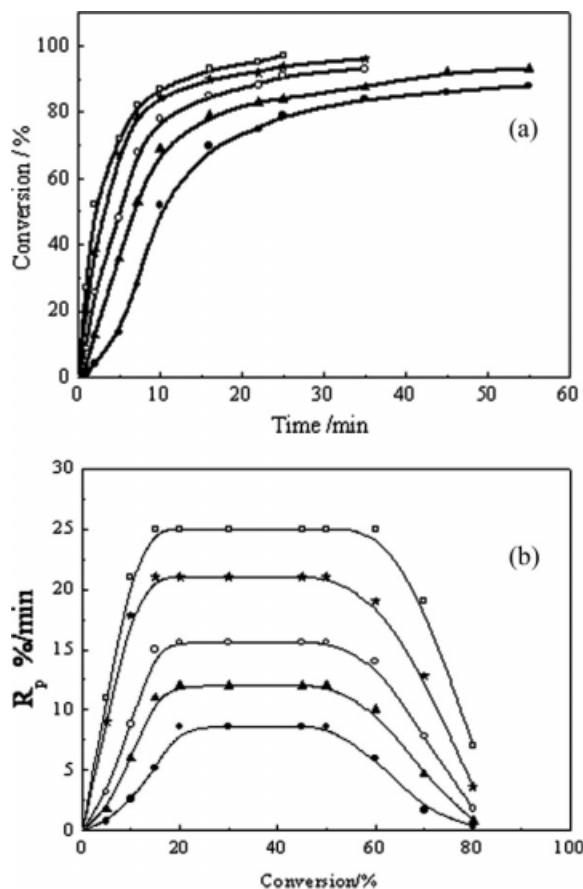


Figure 3 (a) The curves of conversion versus time of MMA polymerization of network I (seed latex) at different temperatures and OP-10(0.025 M), DVB (2%): ●, 65°C; ▲, 70°C; ○, 75°C; ★, 80°C; □, 85°C. (b) The curves of polymerization rate versus conversion of MMA polymerization of network I (seed latex) at different temperatures and OP-10(0.025 M), DVB (2%): ●, 65°C; ▲, 70°C; ○, 75°C; ★, 80°C; □, 85°C.

of emulsion polymerization. The curve of the polymerization rate versus conversion consists of three intervals that are clearly an increasing-rate stage, a constant-rate stage and a decreasing-rate stage. The reason for this is due to the fact that the polymerization of network I is the same as in traditional emulsion polymerization. In addition, the duration of the increasing-rate stage is reduced with the increase in the polymeric temperature.

Polymerization kinetics of network II (PMMA/POA)

The seed latex was produced according to the aforementioned polymerization method but without extraction of samples. The mixture of monomer II containing crosslinking agent was added into the seed latex to swell for an hour. Monomer II is easily diffused into the particles of seed latex via aqueous phase due to their compatibility. The polymerization was carried out at constant temperature after the ini-

tiator was added into system. The kinetic curve of polymerization conversion versus times at different temperatures is shown in Figure 4(a). Through the differential calculation from the curve in Figure 4(a), the relationship of polymerization rate (R_p) to conversion for PMMA/POA(networks II) is shown in Figure 4(b). It can be seen that the polymerization rate at the initial stage is rather high and remains constant up to 70% conversion when the polymerization rate decreases gradually. This indicates that the reaction of network II is fundamentally carried out in the seed latex particles due to the existence of seed emulsion.³⁶ The kinetic curves of network II only show a constant-rate stage and a decreasing-rate stage with no increasing-rate stage. This demonstrates that network II has no fresh nucleation in the second polymerization stage. In the presence of seed latex (network I), monomers of network II are easily

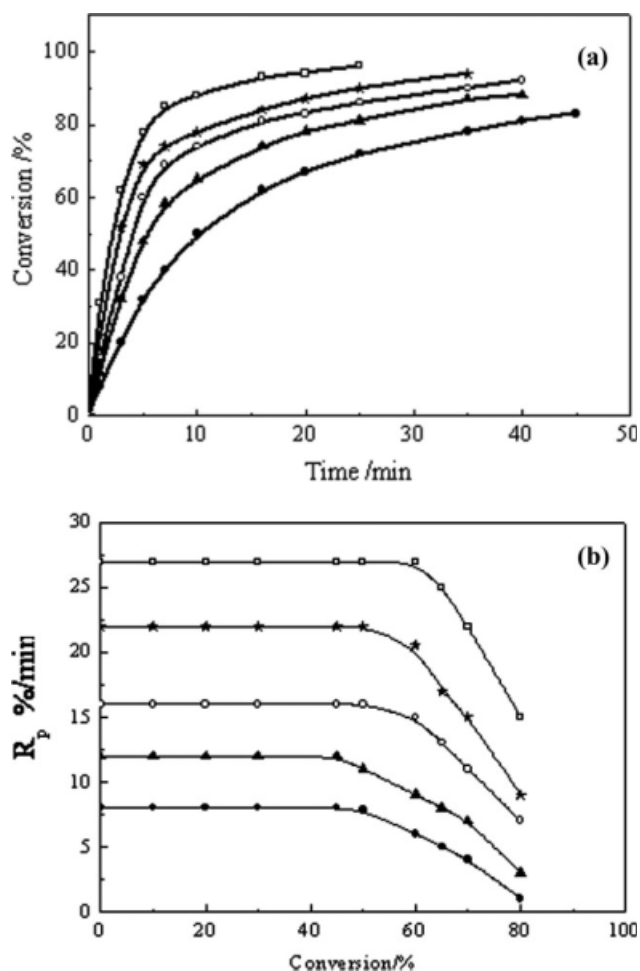


Figure 4 (a) The curves of conversion versus time of MMA/OA polymerization of network II at different temperatures and OP-10(0.025 M), DVB (2%): ●, 65°C; ▲, 70°C; ○, 75°C; ★, 80°C; □, 85°C. (b) The curves of polymerization rate versus conversion of MMA/OA polymerization of network II at different temperatures and OP-10(0.025 M), DVB (2%): ●, 65°C; ▲, 70°C; ○, 75°C; ★, 80°C; □, 85°C.

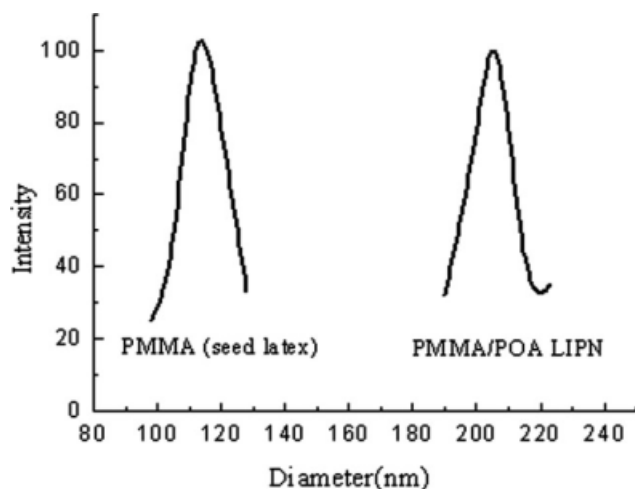


Figure 5 The particle size and size distribution of PMMA(seed latex) and PMMA/POA LIPN (network II) latex.

diffused into the seed latex particles due to their perfect compatibility. Consequently, PMMA/POA LIPN is prepared by the sequential *in situ* polymerization of the second monomer (OA) in the seed latex particles (PMMA). No additional surfactant added to the network II polymerization would avoid fresh nucleation of the second monomer.⁴³

To research the rules of polymerization kinetics of LIPN, we synthesized POA/PMMA LIPN by same method. It was found that the polymerization process of the POA/PMMA (network II) also showed a constant-rate stage and a decreasing-rate stage. The rule of polymerization is the same as for polymerization of MMA/OA.

The particle size and size distributions of PMMA particles and PMMA/POA LIPN particles was shown in Figure 5. Comparing with them, the network II latex particles are clearly larger than the PMMA latex particles. The size distribution of PMMA latex particles is around 120 nm, whereas the size of network II latex particles is around 210 nm and with almost no latex particles with a size of 120 nm. This result indicates that no new particles were produced during the polymerization of network II and the polymerization of network II (POA) was carried out on the seed latex particles (network I PMMA).

Effects of apparent activation energy (E_a) in the process of LIPN polymerization

The influence of temperature on the PMMA reaction rate is shown in Figure 3(b). A linear regression analysis was carried out on the experimental data at the rate-constant stage. According to the Arrhenius equation, the plot $\ln R_p$ versus $1/T$ is illustrated in Figure 6. The apparent activation energy (E_a) of the

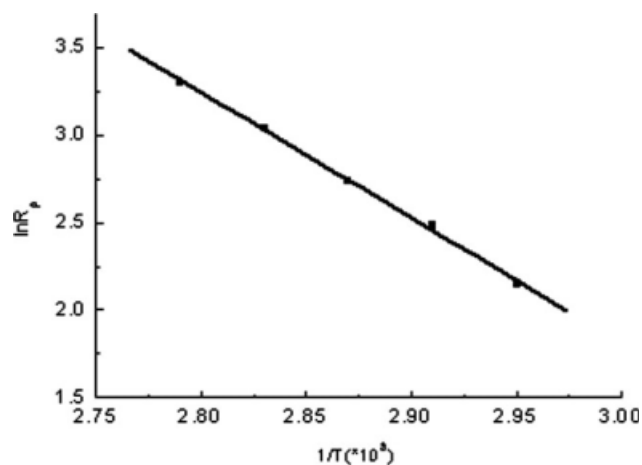


Figure 6 The plot of $\ln R_p$ to $1/T$ of PMMA emulsion polymerization of network I.

PMMA seed latex in the constant rate period was computed to be 61 kJ/mol with a correlation coefficient 0.9981. Using the same method, the curve of $\ln R_p$ versus $1/T$ for PMMA/POA LIPN is shown in Figure 7. The apparent activation energy of PMMA/POA was 60 kJ/mol with a correlation coefficient 0.9973. To compare the E_a values of different LIPNs, POA/PMMA was also synthesized using the same method. The E_a values of network I (seed latex POA) and network II (POA/PMMA) at the constant-rate stage were 62 kJ/mol and 105 kJ/mol, respectively. It is clearly seen that the E_a value (62 kJ/mol) of POA as network I (seed latex) is near to that of PMMA/POA network II (60 kJ/mol) obtained with PMMA as seed latex. But the E_a value (105 kJ/mol) of PMMA as network II with POA as seed latex is higher (approximately 1.7 times) than the E_a value of its self-polymerization PMMA (61 kJ/mol, network I seed latex). We studied the apparent kinetics of polymerization of polyethylacrylate/polystyrene (PEA/PS) LIPN.⁴² The E_a value of PS as network II

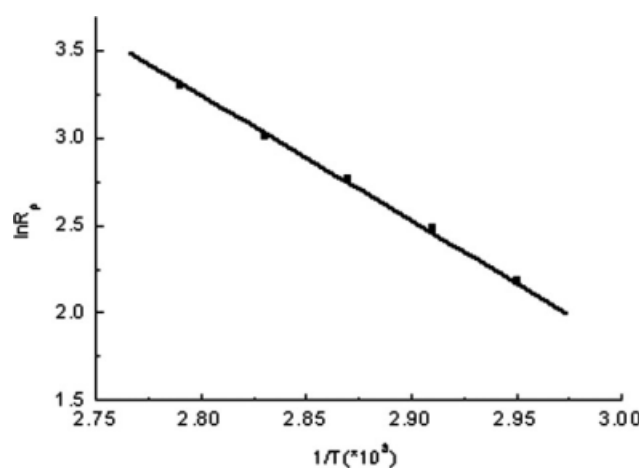


Figure 7 The plot of $\ln R_p$ to $1/T$ for LIPNPMMA/POA emulsion polymerization of network II.

using PEA as seed latex was 80.2 kJ/mol, which is almost twice the E_a value of PS as network I (42 kJ/mol). Dimonie et al.⁴⁴ studied the polymerization of styrene (St) with crosslinking agent in the presence of a kind of elastomer and obtained an E_a value of 88 kJ/mol. It is clear that both PSt and PMMA are glassy polymers, nevertheless, PEA, and POA are rubbery polymers similar to the elastomer. Compared with these results, we found that the polymerization E_a values of PEA, POA as network II with glassy polymer as seed latex are very close to that of self-polymerization. However, as to the monomers (MMA, St, etc.) whose polymers are glassy polymers, the polymerization E_a values of network II with the rubbery polymers (PEA, POA) as seed latex are higher than that of self-polymerization. This indicates that the E_a value of LIPN polymerization presents a clear difference related to the seed latex.

CONCLUSIONS

PMMA/POA LIPN and POA/PMMA LIPN were synthesized using potassium persulfate as initiator and DVB as crosslinking agent. The polymerization rates were found to increase with the increment of OP-10 emulsifier and crosslinker. The polymerization of network I conforms the classical kinetic rules of emulsion polymerization showing an increasing-rate stage, a constant-rate stage and a decreasing-rate stage. But the polymerization of network II only shows a constant-rate stage and a decreasing-rate stage. This demonstrates that network II has no nucleus formation stage and the polymerization of network II occurred in the seed latex particles. The E_a values of network I and network II of PMMA/POA LIPN in the constant-rate interval were 61 kJ/mol and 60 kJ/mol calculated with the Arrhenius equation. Nevertheless, the E_a values of network I and network II of POA/PMMA LIPN were 62 kJ/mol and 105 kJ/mol. The E_a value of POA as network I is near to that of PMMA/POA network II with PMMA as seed latex. But the E_a value of PMMA as network II with POA as seed latex is higher than the E_a value of its self-polymerization PMMA. This indicates that the E_a value of MMA polymerization shows a clear difference related to the properties of the seed latex polymer.

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