Effect of the Chain-Transfer-Agent Content on the Emulsion Polymerization Process and Adhesive Properties of Poly(*n*-butyl acrylate-*co*-acrylic acid) Latexes

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ABSTRACT: The preparation of a series of poly(*n*-butyl acrylate-*co*-acrylic acid) latexes with different contents of chain-transfer agent are reported, and investigations into the adhesive properties of the latexes were made. The latexes were prepared at a solid content of 50 wt % by a two-stage sequential emulsion polymerization with the monomers *n*-butyl acrylate (BA) and acrylic acid (AA). This process consisted of a batch seed stage (BA), which gave a particle diameter of 112 nm, which was then grown by the semicontinuous addition of monomers to a final diameter of 303 nm. Dynamic light scattering was used to monitor the particle diameters and showed that the particles grew without significant secondary nucleation occurring. The molecular parameters were characterized by gel permeation chromatography and dynamic mechanical analysis. The adhesive properties measured were loop tack force, peel strength and shear resistance according to Fédération Internationale des Fabricants et Transformateurs d'Adhésifs et Thermocollants sur Papiers et Autres Supports (FINAT) test methods. The relationship between pressure-sensitive properties and molecular parameters, such as gel content and molecular weight, was evaluated. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 107: 1793–1802, 2008

Key words: adhesion; dynamic light scattering; emulsion polymerization; gel permeation chromatography (GPC); structure-property relations

INTRODUCTION

Pressure-sensitive adhesives (PSAs) are adhesives that remain sticky even when they are dried and cured.[1] The most common products are tapes, labels, and protective films. Although PSAs can be obtained by different polymerization processes (i.e., emulsion, solution, hot-melt, or radiation curing), much attention has recently been devoted to the use of more environmentally friendly processes, such as emulsion polymerization.[2–4] Among different base polymers used in the making of PSAs, high-alkyl acrylates, such as poly(*n*-butyl acrylate) and poly(2-ethyl hexyl acrylate), have enjoyed the fastest growth and the biggest share of the PSA market in commercial applications. However, these applications in PSAs require the formation of a continuous film with high mechanical strength. Both the film formation and mechanical properties of the film depend on the latex particle size and molecular weight as well as on the molecular weight distribution (MWD) of the polymers. Therefore, there is strong incentive to control the particle diameter and molecular weight of the latexes.[5,6] Chain-transfer agents (CTAs) are commonly used to achieve this goal when the latex particle diameters retain the same value. Although a large number of chemicals can act as CTAs, generally, the most used are substances from the mercaptans. The addition of a CTA is not only used to control the polymer molecular weight but may also delay the onset of the gel effect and, in specific cases, introduce some functionality to the polymer end groups. Mercaptans are considered volatile organic compounds and have an offensive odor. Thus, the

amount of CTA in a PSA formulation should be carefully chosen.

In this study, we focused on investigating the semicontinuous emulsion polymerization of *n*-butyl acrylate (BA) and acrylic acid (AA) with a CTA [*tert*-dodecyl mercaptan (TDM)] in a concentration range similar to that used in industrial conditions (≤0.15% on the basis of the monomer weight). The effects of the addition of CTA (TDM) on the gel fraction, entanglement molecular weight (M_e), and soluble molecular weight of poly(*n*-butyl acrylate-*co*-acrylic acid) [poly(BA-*co*-AA)] produced in a semicontinuous emulsion polymerization were studied. Adhesive properties are related to the adhesive performance, such as loop tack, peel force, and shear resistance.

EXPERIMENTAL

Materials

The initiator potassium persulfate (KPS) was obtained from Tianjin Chemistry Agent Co. (Tianjin City, China). The anionic surfactant used in this study was Aerosol MA Series from Cytec, Inc. (Hevens City, The Netherlands) CTA (TDM) was obtained from Merck-Schuchardt (Hohenbrunn, Germany). All these materials were used without further purification. BA and AA were commercial grade and were available from Beijing Dongfang Chemical Co. (Beijing, China). BA monomer was first washed three times with a 2% NaOH solution, then washed with deionized water until the wash waters were neutral, and finally dried with $CaCl_2$ overnight, after which it was distilled under reduced pressure. AA was distilled under reduced pressure before use. Hydroquinone (99%) was used as an inhibitor of the latexes taken from the emulsion polymerization procedure. Deionized water was used throughout the study.

Semicontinuous emulsion polymerization process

PSAs were prepared as 50% solid latexes by semicontinuous emulsion polymerization involving two sequential stages. The first seed stage involved the formation of seed particles 112 nm in diameter with a batch process. This was followed by the second growth stage, which took the final particle diameter to 303 nm. In the second growth stage, various contents of TDM were added to the polymerization system with semicontinuous emulsion polymerization. The surfactant (2.5 g) and water (700.0 g) were added to a 3-L flanged reaction flask. The flow of nitrogen was begun, and the water batch temperature was attained at 78°C. During the following 30 min, the seed-stage BA monomer (50.0 g, 5 wt % of total monomer) was added to the surfactant solution and stirred for 10 min before KPS (2.15 g) dissolved

in water (100.0 g) was added to start the reaction. In the growth stage, the preemulsified monomer mixture of BA (922.64 g) and AA (27.36 g; 95 mol % BA : 5 mol % AA) with surfactant (11.88 g) and weighed TDM from 0 to 0.15% on the basis of the monomer weight were dropped into the flask at a constant rate over 3 h. KPS (0.215 g) dissolved in distilled water (50.0 g) was added to the reaction flask at 115, 175, and 235 min. After the completion of the addition of the growth-stage reactant mixture, a further 60 min was allowed before the latex was cooled to room temperature and filtered through a 53-μm sieve to obtain the coagulate content. Residual monomer was measured with gas chromatograph/mass spectrometer (GC/MS) and was less than 0.1% on the basis of the wet latex weight.

Conversion and particle size measurement of the latexes

At 30-min intervals, samples of the latex (10 mL) were removed into preweighed vials; these were surrounded by ice to quench the polymerization and then analyzed gravimetrically to determine the instantaneous conversion (on the basis of the monomer fed until the sampling time) and overall conversion (on the basis of the monomer fed in the full emulsion polymerization process). Particles sizes were measured at 633 nm with a fixed 90° scattering angle with dynamic light scattering (DLS) on a Malvern Zetasizer (Worcestershire, UK) 3000HS, and the cell temperature was controlled at 25 ± 0.1°C. The particle diameters quoted are the mean values of the *z*-average diameters (d_z's) calculated by the cumulants method.

Solvent extraction and molecular weight characterizations

The soluble polymer fractions were separated from poly(BA-*co*-AA) polymers by extraction of the dried latexes with boiling tetrahydrofuran (THF) for 2 days by means of Soxhlet extraction. The insoluble polymers left in the thimble were dried in a vacuum oven at 60°C for 48 h to obtain the gel content, which was considered the ratio of the insoluble polymer content to the initial polymer content by weight. The soluble polymers were subjected to molecular weight measurements with an apparatus (Waters 515, Milford, MA) equipped with refractive-index detector (Waters 2410) and data system (Millennium 32). THF was used as the flow phase. A calibration curve was constructed from polystyrene standards.

Dynamic mechanical analysis (DMA)

Dynamic mechanical properties, including the storage modulus (G') and damping factor (tan δ) of poly(BA-*co*-AA), were obtained with a Triton (Key-

worth, UK) 2000 dynamic mechanical analyzer in plate clamp mode. The plate sample with typical dimensions of 10 × 5 × 2 mm^3 was prepared through cast molding. The heating rate and frequency were 5°C/min and 1 Hz, respectively. Glass-transition temperatures (T_g's) were located from the peaks in the loss tangent.

PSA testing

The latexes prepared were adjusted to pH 5.5 with a 25 wt % ammonia solution and filtered again. After that, they were coated with a Elcometer (Manchester, UK) 4360/15 bar onto 36 μm thick poly(ethylene terephthalate) to give a film with a dry thickness of 30 μm and dried at 105°C for 4 min. Adhesive bonds were formed by the application of a standard 2-kg roller passing over twice. All adhesive testing was performed at 23°C and 50% relative humidity, and the samples were climatized into this condition 24 h before testing. Loop tack and 180° peel were done off a stainless steel substrate. Test methods were in accordance with the Fédération Internationale des Fabricants Transformateurs d'Adhésifs et Thermocollants sur Papiers et Autres Supports (FINAT) test methods numbers 9 and 1 at 300 mm/min. Shear resistance was done off a glass plate substrate with a 25 × 25 mm^2 poly(ethylene terephthalate) coated strip and a 1000-g hanging weight according to FINAT test method number 8. The average values were from five trials.

RESULTS AND DISCUSSION

Latex preparations

Latexes were prepared with seeded emulsion polymerization (i.e., the addition of monomer, initiator, and surfactant to a previously prepared latex), which had the advantage of preventing the uncertainties of the particle initiation stage and, therefore gave better batch-to-batch reproducibility. Instantaneous and overall conversions were calculated from a mass balance of the reagents in the polymerization with the percentage solid content measured at each sampling time:

Instantaneous conversion (%)

$$= \left(\frac{\text{Mass of polymer formed}}{\text{Mass of monomer added}}\right) \times 100$$

where the mass of monomer added is the sum of the monomer in the seed stage and any monomer that has been added during the growth stage:

Overall conversion (%)

$$= \left(\frac{\text{Mass of polymer formed}}{\text{Total mass of monomer}}\right) \times 100$$

where the total mass of monomer is the sum of the monomer in the seed stage and all of the monomer in the growth stage.

Plots of conversion versus reaction time for each of the latex preparations are shown in Figure 1. All of the polymerizations were observed to proceed at high instantaneous conversion (>85%), that is, under monomer-starved conditions. Final overall conversions were found to be high (>99%) for all of the polymerizations, which showed that a continuation of the polymerization for 1 h after the end of the monomer addition stage was adequate to allow for complete conversion.

The DLS technique was used to obtain quantitative information about the particle sizes of colloidal systems. In this study, DLS provided a rapid means of monitoring the particle size of the latex particles during both the seed and growth stages of polymerization. With this information, it was possible not only to establish and reproduce a latex system of known particle diameter but also to determine whether, during the growth stage of polymerizations, the latex particles grew sequentially or if secondary nucleation occurred.

Latex particle diameters were determined by DLS and compared with those theoretically calculated from the following equation:

$$d_t = \left(\frac{M_t I}{M_s}\right)^{\frac{1}{3}} \times d_s$$

where d_t is the diameter of the particle at time t, M_t is total mass of the monomer added at time t, I is the instantaneous conversion, M_s is mass of monomer added in the seed stage, and d_s is the seed particle diameter as measured by DLS. The density of poly(BA-*co*-AA) was assumed to be the same as that of poly(*n*-butyl acrylate) (PBA). Plots of the particle diameter versus reaction time for each of the latex preparations are shown in Figure 2. The good agreement shown between the experimental and theoretical particle diameters throughout the polymerization for all of the latexes provided strong evidence that the observed particles were grown without significant secondary nucleation and that all of the polymer particles formed were spherical. This, coupled with the low levels of coagulum (<0.5%) measured for all of the latexes, showed that the correct surfactant concentration was used in the growth stage of the polymerization. The final overall conversions of nearly 100% were achieved with a 50% solid content with final particle diameters of 303 ± 3 nm.

The final values for the latexes comprising various TDM contents in the second growth stage are listed in Table I. The particle sizes for all of the latexes were almost the same within the experimental error. At different levels of TDM content, the colloidal stability of the latexes was not affected because the coagulum levels for these five latex preparations were below 0.5 wt %. Therefore, the presence of CTA had no
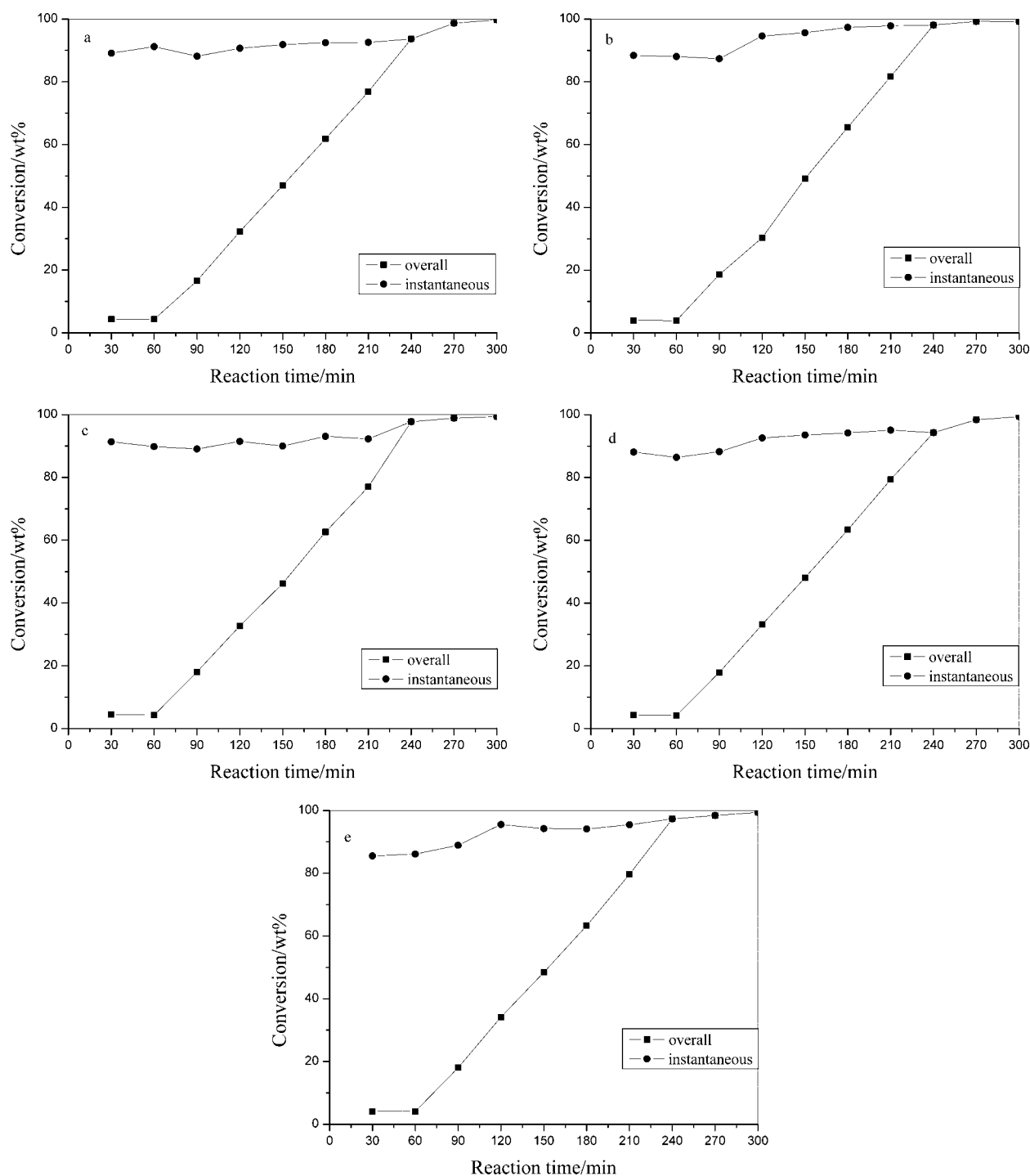
Figure 1 Variation of the overall and instantaneous percentage conversions with the reaction time with different CTA contents: (a) 0, (b) 0.035, (c) 0.07, (d) 0.10, and (e) 0.15%.

significant effect on the final monomer conversion and the particle size in the emulsion polymerization.

Molecular characterization

Gel content

After the latex was dialyzed for 7 days to remove the surfactant, coagulated samples were obtained by

freeze–thaw cycling and washed with deionized water several times. After the samples were dried, the gel contents of the copolymers were determined by Soxhlet extraction. The gel arose from termination by the coupling of propagating long-chain branches formed by intermolecular chain transfer to polymer. Figure 3 shows the values for the gel fraction at different CTA contents. According to the previous
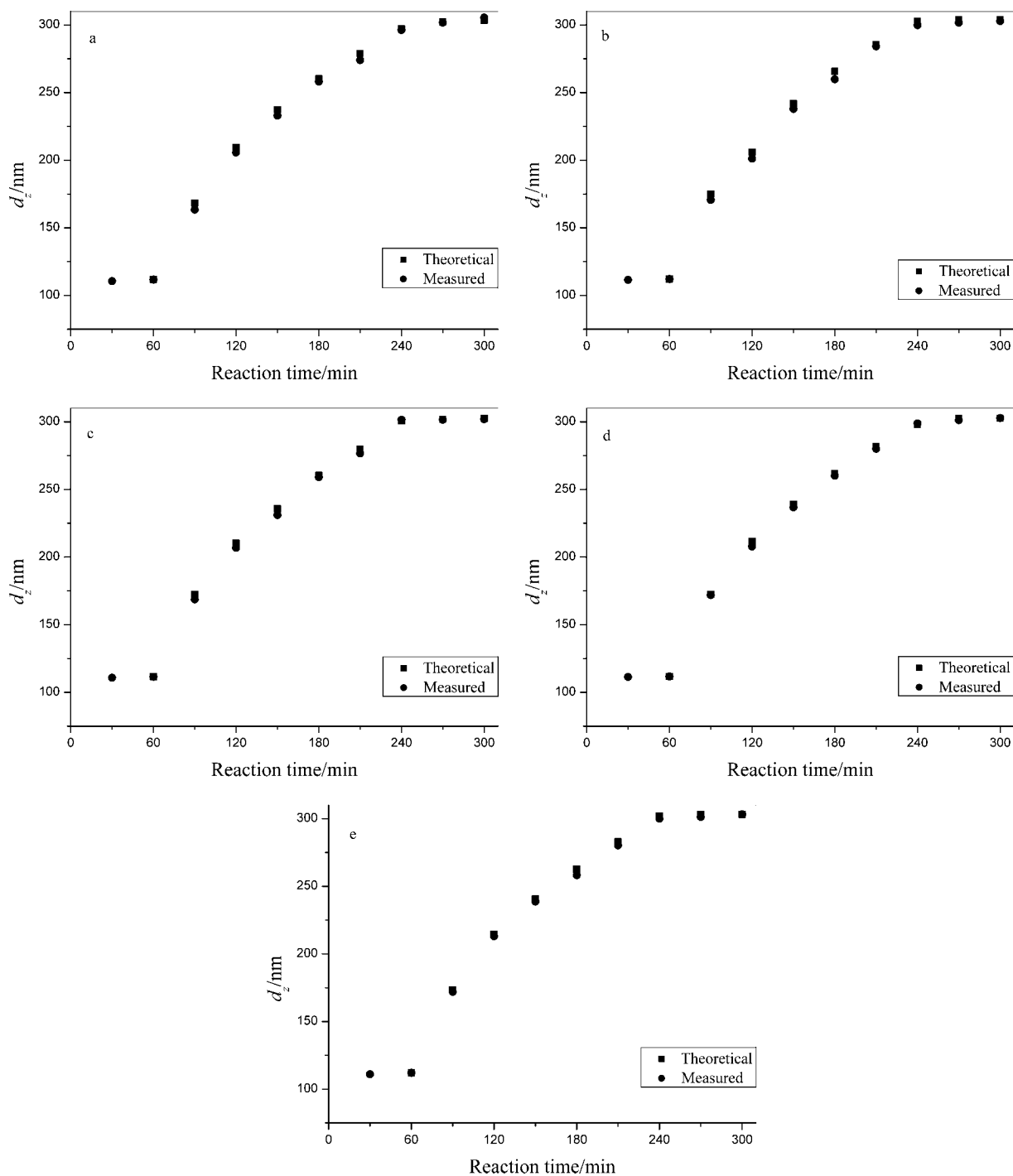
Figure 2 Variation of the measured and theoretical d_z with the reaction time with different CTA contents: (a) 0, (b) 0.035, (c) 0.07, (d) 0.10, and (e) 0.15%.

results, the emulsion polymerization of poly(BA-*co*-AA) latexes prepared was run under monomer-starved conditions. Thus, the polymer concentration in the latex particles was high at all times, and as a result, microgels were formed early. As the reaction proceeded, the formation of microgels increased and the molecular weight of the THF-soluble fraction

TABLE I
Summary of the Final Data for the Poly(BA-*co*-AA) Latexes

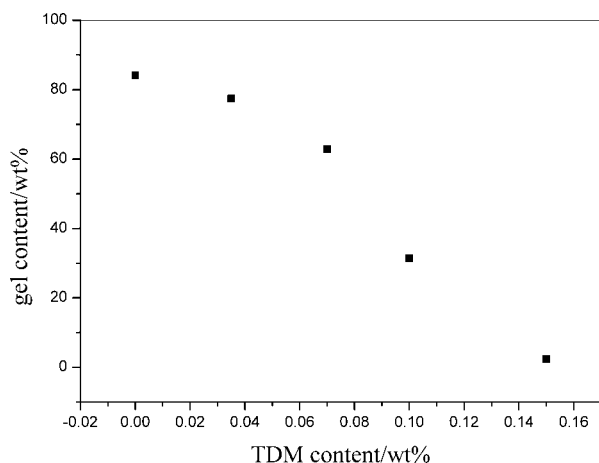| TDM content (%)	0	0.035	0.07	0.10	0.15
Final conversion (%)	99.8	99.2	99.4	99.4	99.4
Final particle size (nm)	306	303	302	303	303
Coagulate content (%)	0.11	0.47	0.44	0.46	0.42

Figure 3 Variation of the gel content with the TDM content.

decreased.[7] This was expected because higher molecular weight species are the most prone to hydrogen abstraction and chain transfer because of the higher number of tertiary carbon atoms per chain.[8] The higher the CTA concentration was, the lower the gel fraction was. With the addition of 0.15% CTA, the gel fraction was almost completely avoided. The presence of modest amounts of TDM made the chain transfer to TDM the main termination event. Under these circumstances, the kinetic chain length was considerably reduced, and the role of the termination by combination was not as important. Therefore, the gel fraction was significantly decreased.

Molecular weight

Table II presents the evolution of the soluble polymer molecular weights and their distribution. The final soluble polymer molecular weight decreased slightly as the amount of the TDM used in the experiment increased. The TDM content had no significant effect on the soluble MWD. The reason for this behavior was that the mechanism leading to the decrease of the soluble molecular weight was not the same throughout the series. In the experiments carried out without and with 0.035 wt % TDM, the decrease of the soluble polymer molecular weight was due to a transfer from the soluble fraction to the gel of large and branched polymer chains formed by chain transfer to the polymer plus termination by combination reactions. On the contrary, in the experiments carried out with larger amounts of TDM (0.10 and 0.15 wt %), the decrease in molecular weight was mainly due to a decrease in the kinetic chain length by chain transfer to the TDM. In these experiments, almost no gel was formed, so the transfer of large molecules from the soluble fraction to the gel was not significant.

Transitional behavior

DMA is a sensitive thermal analytical technique for the detection of transitions associated with molecular motions within polymers in the bulk state. In Figure 4, the dynamic mechanical spectra of the films cast from the latexes show only one transition. The width of the glass transition was related to the properties of these polymers. A simple sharp transition occurred in the tan δ at T_g for high TDM contents. When the TDM content decreased, as shown in Figure 4, the transition regions were much broader than those observed for the high TDM contents. The broad nature of the glass transition was a result of the broad range of copolymer composition. When the transfer to the CTA reaction was the dominant termination mechanism, this yielded a polymer with a homogeneous composition and a constant molecular weight. T_g's of the poly(BA-co-AA) polymers with different TDM contents in the growth stage are given in Table III. The T_g's measured by DMA were $-25 \pm 1°C$, and no significant decrease in T_g was observed with increasing the level of CTA. This was because the polymer produced during the emulsion polymerization was of the same initial monomer feed composition.

Another utility of the DMA data is the determination of M_e. M_e can be estimated from the rubbery plateau modulus (G_N^o), shown as follows:

$$M_e = \frac{\rho_p R T}{G_N^o}$$

where ρ_p is the density of the polymer, R is the gas constant and is equal to 8.31×10^7 dyne cm mol^{-1} K^{-1}, T is the absolute temperature where G_N^o is located, and G_N^o is determined from G' at the onset of the rubbery region (usually where tan δ reaches a minimum after a prominent maximum). For crosslinked PSA, it was determined as a point of inflection in tan δ curve following the prominent maximum. The results of M_e values are listed in Table III also.

Adhesive properties

The adhesive testing results are shown in Figure 5. Increasing the TDM content in the second growth

TABLE II
Summary of the Molecular Weights of the Soluble Poly(BA-co-AA) Polymer

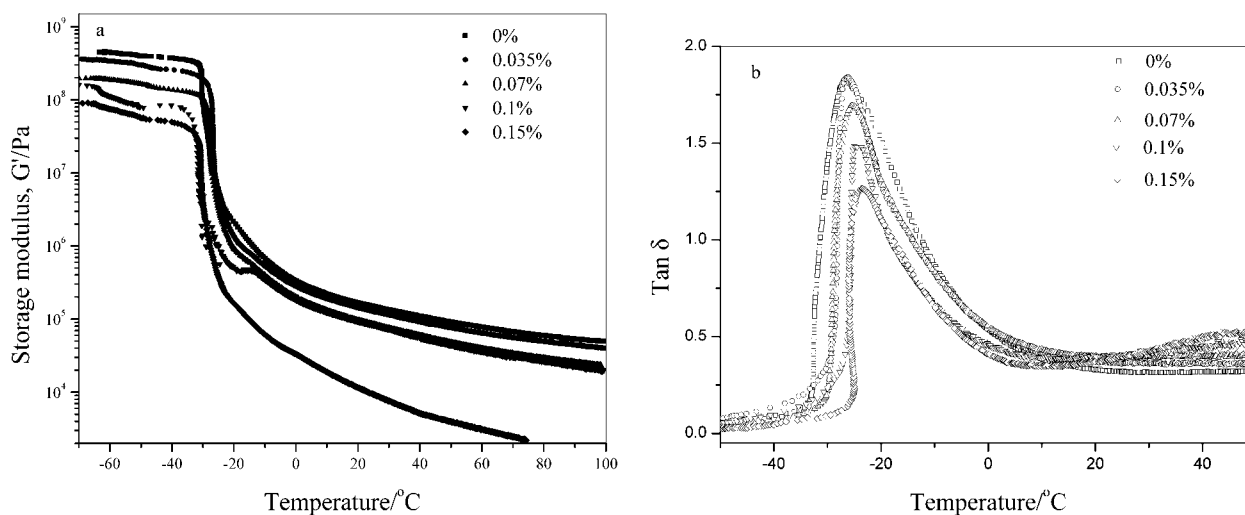| TDM content (%)	0	0.035	0.07	0.10	0.15
M_n (g/mol) $\times 10^{-4}$	8.1	7.8	6.8	6.1	5.6
M_w (g/mol) $\times 10^{-4}$	31.6	30.2	25.4	23.6	20.1
MWD	3.90	3.87	3.74	3.87	3.59

Figure 4 Curves of (a) G' and (b) tan δ versus the temperature of the PSA polymers with different CTA contents.

stage from 0 to 0.15 wt % served to increase the loop tack force and peel strength but reduced the shear resistance, almost to zero (10 min) at 0.15%TDM content.

The shear resistance of an adhesive is that which resists the tendency to flow or creep. This property is of great importance in PSA applications. The mechanism of bond failure must be in the bulk of the adhesive and not at the interface for the test to be a measure of cohesive strength. As shown in Figure 5(a), coated PSAs showed a dramatic decrease in shear resistance as the TDM content increased, with the mode of failure being cohesive for all tests. Emulsion polymerization of low-T_g acrylics carried out to complete conversion produced a significant amount of microgels inside the particles due to chain transfer to the polymer via the hydrogen abstraction of tertiary vinyl carbons. The gel content and molecular weight of the soluble polymers decreased as the TDM content increased. The higher the gel fraction was, the higher the crosslinking densities were. Meanwhile, a reduction in molar mass reduced the level of chain entanglement in the bulk polymer, as shown in Table III, where the M_e values increased with increasing TDM content. When the TDM was low, the chain ends from the micronetworks could entangle with the soluble polymer chains, which, in turn, could entangle with other chain ends from another particle after film formation. The micronetworks were no longer entangling with the soluble polymer chains as the M_e values increased when the TDM contents were over 0.1%. Chain entanglements behaved as pseudocrosslinks that eventually disentangled under shear stress but contributed to the measured shear resistance.

Shear resistance, or time to failure (T), is directly proportional to zero-shear viscosity (η), as given by the following equation:[9]

$$T = \frac{L^2 W \eta}{2tMg}$$

where L is the length of overlap, W is the width of the tape, t is the adhesive thickness, M is the load, and g is the gravitational constant. When the TDM content increased, the molecular weight of the soluble polymers decreased, and the percentage fraction increased. The soluble polymers with low molecular weights acted as plasticizers to decrease zero-shear viscosity. Otherwise, because of the absence of chemical crosslinking, they showed poor cohesive strength when the TDM content reached 0.15%. Therefore, gel content and network morphology played a significant role in influencing the shear resistance.

The force measured during peel was composed of two components. First, the force required an overcoming of the work of adhesion, that is, a breaking of the adhesive/adherent interfacial bond, and second, the force required the deformation of the bulk of the adhesive.[10,11] The peel was the outcome of the viscoelastic process. It bore a direct relation to the molecular weight of the polymers. Figure 5(b) shows the relationship between the peel force and TDM content. As shown by the results of Table II, the soluble polymers with broad polydispersity values (MWD = 3.61–3.89) were effective in providing high-viscoelastic-energy dissipation during peeling. Without or with low contents of TDM, high weight-

TABLE III
Values of T_g and M_e of the Poly(BA-*co*-AA) Polymer

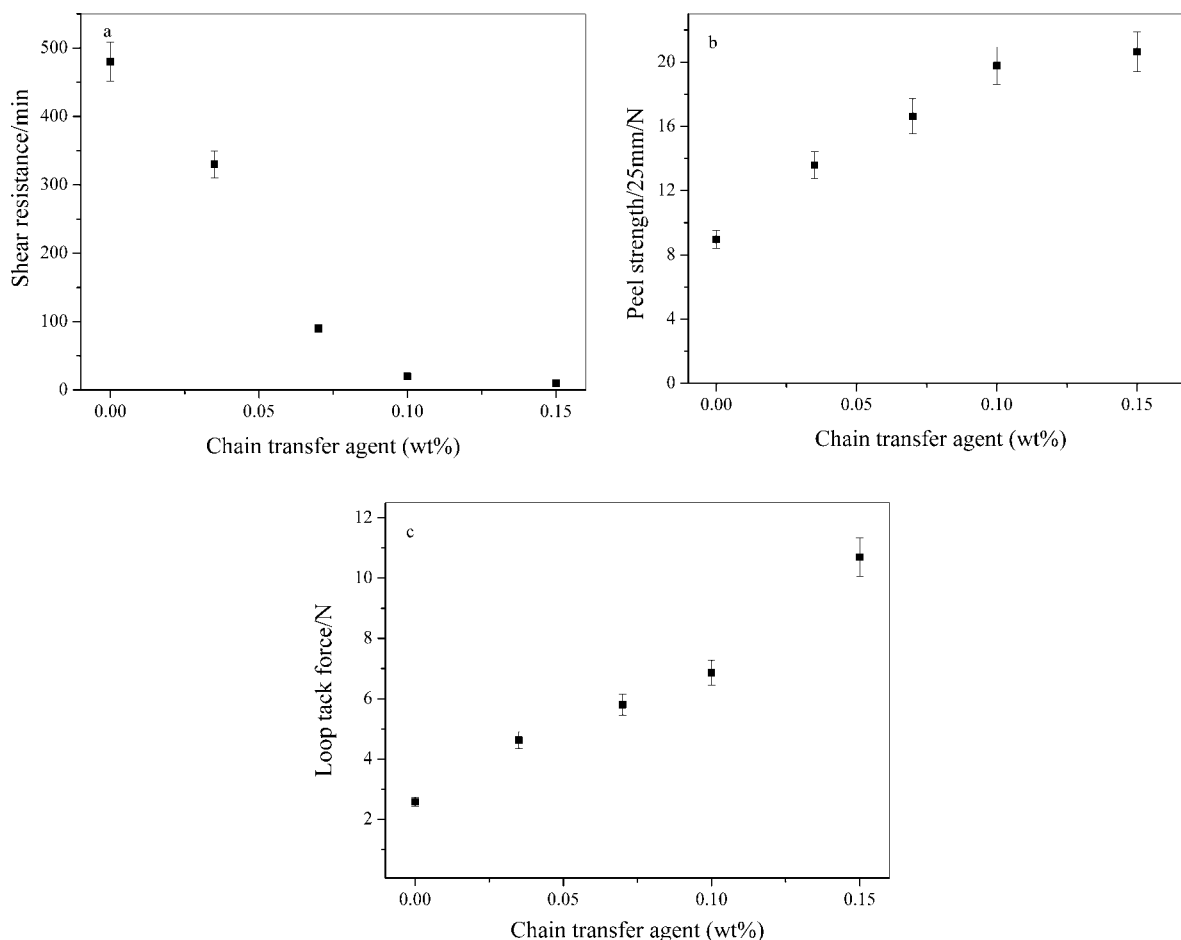| TDM content (%)	0	0.035	0.07	0.10	0.15
T_g (°C)	−24.4	−25.0	−25.3	−25.6	−26.1
M_e (g/mol) × 10^{-4}	14.1	20.0	23.2	28.3	34.9

Figure 5 Effects of the TDM content on the adhesive properties: (a) shear resistance, (b) peel strength, and (c) loop tack force.

average molecular weight (M_w), especially when the number-average molecular weight (M_n) was also high, gave less viscous flow during bonding and more elastic response during debonding. Therefore, it did not allow any mobility for the polymer chains. As a result, the resistance to peel was low. The addition of a small amount of TDM substantially improved adhesion to the substrate plate. A high molecular weight was needed for the PSA film to undergo fibrillation during the peeling process. This was observed experimentally with the polymer forming elongated filaments, which significantly increased peel energy.

Tack, which is inversely proportional to the elasticity modulus, should be high enough that the adhesive surface achieves bonding from merely contacting and pressing to a substrate. The tack also provides resistance of an adhesive film to detachment from a substrate.[12,13]

It is known that the tack of a polymer is decreased by crosslinking. With increasing crosslink density, the ultimate strain decreased. This response was

more elastic during debonding. The DMA data (Fig. 4) showed an identically single T_g, which indicated that homogeneous films were easily formed because of fast diffusing species from the soluble fraction of the polymers. The strength of an adhesive bond is determined by the thermodynamic contributions to the interfacial energy (van der Waals interactions, electrostatic forces, and hydrogen bonding) and the rheological contributions due to the viscoelastic dissipation during deformation of the polymer chains in the adhesive layer itself. In a tack test, the work of adhesion is dominated by this viscoelastic contribution. For the high molecular weights from the soluble polymers, fibrillation increased the work of adhesion above that due to the contributions of good initial wetting and initial resistance to flow. In the tack measurement of the high debonding rate, the polymer chain mobility was sufficiently high to allow the formation of an interfacial bond that exceeded the strength of the bulk polymer. So, the loop tack force increased with increasing CTA content, as shown in Figure 5(c). Hence, high visco-

elastic energy dissipation could be obtained when there was good anchorage of the adhesive onto the substrate and low modulus–high elongation fibrils that were deformed during the debonding process.

Effect of the peel test rate on the peel strength

To test the effect of the mode of failure during peeling on the peel strength, a series of peel tests were conducted at different peel rates. Peel rates from 5 to 500 mm/min were used, including the standard peel test rate of 300 mm/min. Figure 6 shows graphically the relationship between the peel force and peel rate for the latexes with different TDM contents, and Table IV lists the mode of failure after the peel test.

For the same content of TDM, peel forces increased at low peel rates, revealed the cohesive failure, and then leveled off with the failure of adhesion at high rates. According to the previous discussion, the cohesive strengths of the PSA films decreased with increasing TDM content under conditions of static shear loading. However, with increasing peel rate, the modulus of the PSA film increased with the time–temperature equivalent principle; that is, the viscous response of the polymers at the low peel rate transferred to the rubbery response at the high peeling rate.[14] The measurement of peel force was equivalent to that at low temperatures with the increasing peel rates.

Peel force passed through a maximum at the different peel rates, which occurred at approximately 100 mm/min for 0 and 0.035% TDM contents, at approximately 200 mm/min for 0.07 and 0.1% TDM contents and approximately 300 mm/min for 0.15% TDM content. These results correspond to the failure mode as shown in Table IV from cohesion to interfa-
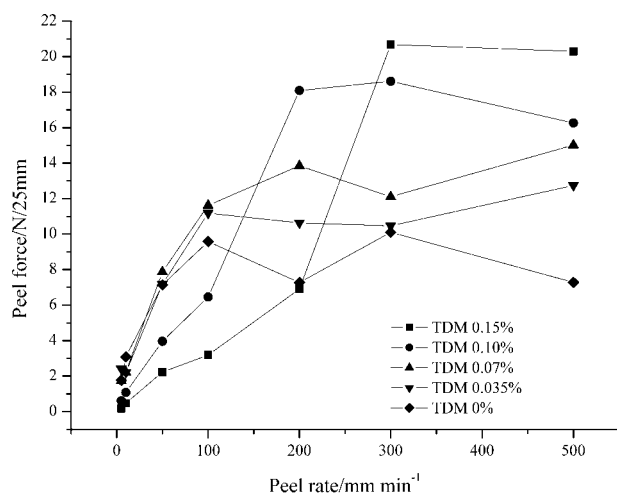
TABLE IV
Effect of the Peel Rate on the PSA Failure Mode with Different TDM Contents

Peel rate (mm/min)	TDM content (%)				
	0	0.035	0.07	0.10	0.15
5	○	○	○	○	○
10	○	○	○	○	○
50	○	○	○	○	○
100	○/●	○/●	○	○	○
200	●	○/●	○/●	○/●	○
300	●	●	●	○/●	○/●
500	●	●	●	●	○/●

○ = cohesive failure; ● = adhesive failure.

cial adhesion at the specified peel rates. This was the reason why only peel rates up to 300 mm/min were taken into consideration in the FINAT test method.

CONCLUSIONS

DLS was used to monitor the growth of particles during polymerizations with different TDM contents at the growth stage. Comparison of theoretically calculated particle diameters with those measured experimentally showed that the particles grew without significant secondary nucleation. Gravimetric analysis was used to measure the conversion of the monomer to polymer throughout the reactions and revealed that the polymerizations proceeded under monomer-starved conditions (instantaneous conversion > 85%). An increase in the level of CTA at the growth stage from 0.0 to 0.15 wt % to monomer caused the shear resistance to decrease and the peel force and loop tack to increase. The amount of gel polymer clearly played an important role in the shear resistance, whereas the peel force and loop tack were mainly affected by the molecular properties of the soluble polymers in PSAs. A higher soluble fraction led to an increase in the loop tack and peel force because of increasing viscoelastic energy dissipation and more contact with the substrate. A series of peel tests were conducted at peel rates varying from 5 to 500 mm/min. At low peel rates, the PSAs exhibited cohesive failure, whereas at high rates, the PSA polymers failed adhesively. This behavior was attributed to the viscous response to rubbery one.

Figure 6 Variation of the PSA peel force with different peel rates.

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