

Synthesis of Acrylate Polymers by a Novel Emulsion Polymerization for Adhesive Applications

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ABSTRACT: Pressure-sensitive adhesives (PSAs) are viscoelastic–elastomeric materials that can adhere strongly to solid surfaces with light contact pressure and a short contact time. Polyacrylates produced by solution polymerization are used widely because of their good adhesive properties. A novel emulsion polymerization was established to improve the low physical properties of PSA on the basis of conventional poly(*n*-butyl acrylate) (PBA) by emulsion polymerization. PBA latex was synthesized by the emulsion polymerization of 50 wt % *n*-butyl acrylate mixed with 15 wt % ethyl acetate (EA) with Emal-10P and Emulgen-920 as anionic and nonionic surfactants, respectively, at 70°C. Potassium persulfate (KPS) or a combination of KPS and dicumyl peroxide (DCP) was used as the initiator. The KPS/DCP system gave a very high-molecular-weight PBA of a narrow molecular weight distribution with a weight-average molecular weight/number-average molecular weight value of 1.01–

1.03 in 15 min. The PSA tape was prepared by the casting of the PSA latex onto a corona poly(ethylene terephthalate) film as an adherent to obtain a 50- μ m-dry-thickness film. The PSA tape produced from PBA by the novel emulsion polymerization showed better adhesive properties, such as 180° peel adhesion, shear holding power, and rolling ball tack tests according to JIS and ASTM standards, than PSA tape produced from solution polymerization. The occlusion of a small amount of EA in emulsion particles before polymerization was found to give higher properties than those of PBA prepared by the addition of EA to the PBA latex after polymerization. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 100: 413–421, 2006

Key words: adhesives; emulsion polymerization; acrylate polymer

INTRODUCTION

The term *pressure-sensitive* or sometimes *self-sensitive* is used to designate a distinct category of adhesives. Pressure-sensitive adhesives (PSAs) are viscoelastic–elastomeric materials that can adhere strongly to solid surfaces upon the application of light contact and a short contact time. PSAs are different from viscous liquids, in that the typical peel force required to peel a PSA tape off a substrate can range from 1 to 10 kg/cm², whereas a tape coated with a viscous liquid such as honey or molasses would take less than 0.1 kg/cm².¹ PSAs are aggressively and permanently tacky in their dry form, are solvent-free at room temperature, and firmly adhere to a variety of dissimilar surfaces. PSAs may be classified according to the physical form in which the adhesive is supplied or according to the chemical composition, that is, the main elastomers used in the adhesive formulation. Although many other polymers are used as the basis for adhesive compounding, the major share falls to natural rubber,

polyacrylate, and block copolymers. However, acrylate polymers possess some properties that are superior to those of many other polymers used for PSAs because polyacrylates of a certain monomer composition are inherently pressure-sensitive without any additional compounding. Their popularity is mainly attributable to their low glass-transition temperature, low plateau modulus, optical clarity, oxidative, ultraviolet, and migration resistance, low toxicity, and low cost.^{2,3} Only a few other polymers, that is, *n*-butyl acrylate (*n*-BA), 2-ethylhexyl acrylate, and isooctyl acrylate, show this character. After the polymerization is completed, the structure of the polymer becomes saturated, and this leads to high resistance to oxidation. It is transparent and colorless and does not yellow upon exposure to sunlight. It shows better resistance to oxidation than other polymers used for PSAs. Acrylate PSAs are produced by either solution or emulsion polymerization.² Polymer solutions in organic solvents are most frequently used for PSA preparation. Solution polymers give a homogeneous adhesive film, which is free from impurities such as surfactants and wetting agents, which are normally necessary in emulsion polymers. Their adhesive properties are superior to those of emulsion adhesives, especially in

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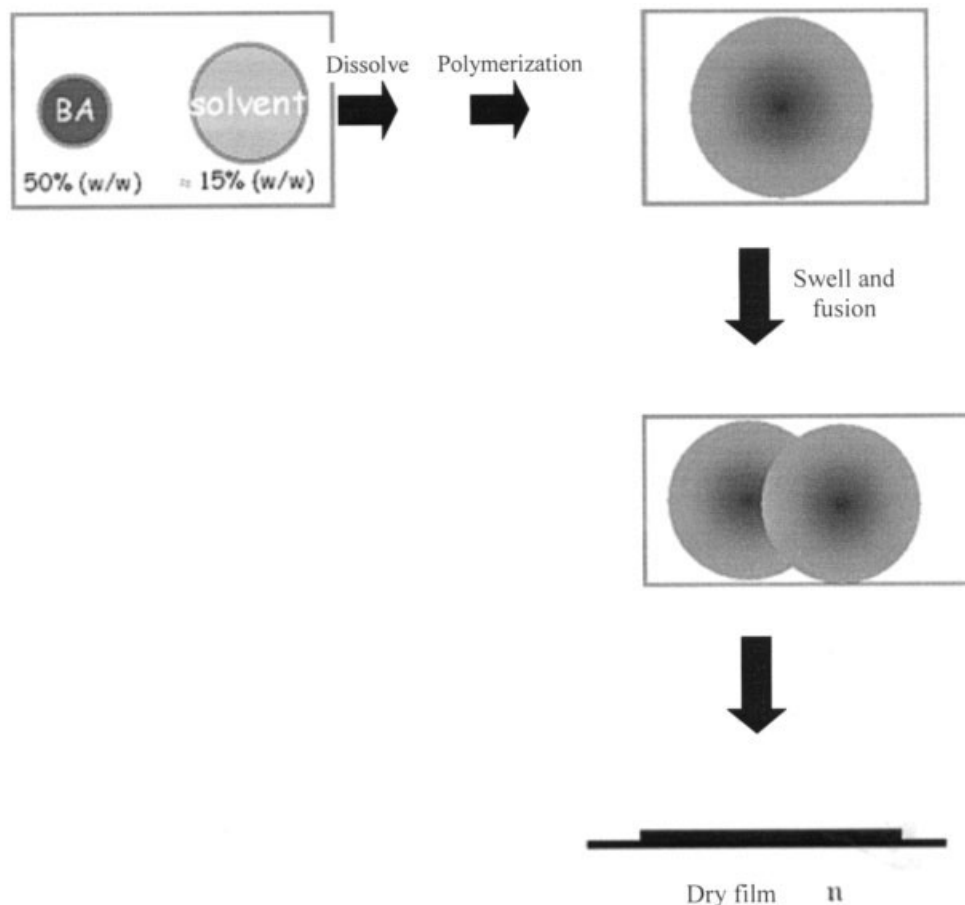


Figure 1 Schematic representation of the fusion of polymer particles by the addition of an organic solvent.

applications for which high shear resistance is required together with good tack and peel strength.^{4,5} This is due to their continuous network morphologies formed by the crosslinking reaction of acrylic acid and aluminum acetyl acetonate in the film, once the solvent has evaporated.^{6,7} Nevertheless, the consumption of solution PSAs has substantially decreased because of environmental restrictions; for example, solvent vapor exhaust into the atmosphere is strictly regulated.² More recently, emulsion polymerization has become by far the most important process for producing acrylate PSAs. Acrylate polymers are environmentally safe, easy to handle, and economical.

The purpose of this work is to elucidate the new polymerization method of poly(*n*-butyl acrylate) (PBA) for PSA applications based on reducing the amount of organic solvents in the solution polymerization. The organic solvent is expected to assist the fusion of polymer particles, resulting in homogeneous film formation on poly(ethylene terephthalate) (PET) as an adherent after the drying of the PSA film, as shown in Figure 1. The inclusion of an organic solvent can be performed by the addition of a solvent to the emulsion of a monomer to give swollen polymer particles after the polymerization has completed. The ad-

dition of an organic solvent to the monomer mixture is expected to have different effects on the properties of PSAs produced from the latex. An emulsion polymerization in the presence of a mixed organic solvent has not been reported. A modified emulsion acrylate requires the production of very small particles of acrylate polymers to make an acrylate PSA film having adhesive properties that are the same as or better than those obtained by normal solution polymerization.

EXPERIMENTAL

Materials

The *n*-BA monomer (analytical-reagent-grade; Fluka) was purified by extraction with 10% (w/v) aqueous sodium hydroxide and distilled *in vacuo* before use. The initiator potassium persulfate (KPS) benzoyl peroxide (BPO), and dicumyl peroxide (DCP) were analytical-reagent-grade and were available from Fluka. The surfactants were Emal-10P and Emulgen-920, and they were obtained from Kao Co., Ltd, (Tokyo, Japan). Analytical-reagent-grade ethyl acetate (EA) and toluene, which was distilled before use, were obtained from BDH (Dor-

set, UK) and Fluka (Hannover, Germany), respectively. Deionized water was used as the polymerization medium.

Preparation of the monomer emulsion

n-BA (50 wt %) as a monomer, 1.5 wt % Emal-10P as an anionic surfactant, 0.5 wt % Emulgen-920 as a nonionic surfactant, and a partial volume of an organic solvent were added to a 500-mL glass reactor, which was heated by an oil bath at 70°C, under a nitrogen atmosphere. The reaction mixture was homogenized with a Heidon homogenizer (Germany) at 7000 rpm for 15 min. The resulting monomer emulsion was milky in appearance.

Preparation of the initiator solution

The initiator solution could be divided into two systems: KPS only and a combination of KPS and DCP, which was performed by the addition of 0.05 wt % initiator, based on the total weight, to deionized water and stirring with a magnetic bar at room temperature for 5 min.

Polymerization procedure

After the monomer emulsion was homogenized for 15 min, the initiator solution was fed slowly into the reactor. With the exotherm taken into account, the batch temperature was maintained at $70 \pm 1^\circ\text{C}$. After all the initiator had been added, the reaction was continued for 180 min when KPS only was used as the initiator and for around 15–30 min when a combination of KPS and DCP was used to decrease the residual monomer.

Latex characterizations

The particle size distribution (PSD) of the latex was analyzed by light scattering with a Mastersizer 2000 (version 3.01, Malvern Instruments, Ltd., Worcestershire, UK). The latex sample was dropped into the sample chamber until the light observation was in the range of 0.5.

The viscosity-average molecular-weight (M_v) was measured with a dilute solution viscosity technique. The PBA solution in acetone [0.05% (w/v)] was filtered through a glass filter (2 G 3) before the measurement. The accurate concentration of the PBA solution was determined from the percentage of dried PBA in 2.00 mL. The viscometric measurements were performed in an Ubbelohde viscometer (model AVS 350, Schott, Germany) placed in a constant-temperature bath controlled at $25.00 \pm 0.01^\circ\text{C}$ and were repeated until five consecutive readings differed by ± 0.02 s. M_v was calculated with the parameters reported in as follows:⁸

$$[\eta] = (6.85 \times 10^{-5})M_v^{0.75}$$

where $[\eta]$ is the intrinsic viscosity.

The resulting PBA prepared with the KPS and DCP combination initiator system could not dissolve in tetrahydrofuran, so it could not be analyzed by gel permeation chromatography. Thus, the solvent fractionation of PBA was applied to the determination of the molecular weight distribution (MWD) and PSD. The dried sample was dissolved in acetone at a concentration of 2% (w/v). The temperature was controlled by a water bath at 20°C. Methanol was slowly dropped into the solution, which was kept stirring until a white precipitate was observed. After that, the temperature was increased to 40°C to redissolve the precipitate. This solution was slowly cooled until the precipitant was observed again and was permitted to stand at 40°C for 24 h. After the precipitate was recovered, the residual solution was subjected to further fractionation in a similar way. Each precipitated sample (ca. seven to eight fractions) was collected and subjected to the determination of M_v . The MWD and PSD were estimated as follows:

$$M_n = \frac{\sum W_i}{\sum N_x}$$

$$N_x = \frac{W_i}{M_v}$$

$$M_w = \frac{\sum W_i M_v}{\sum W_i}$$

$$\text{PSD} = \frac{M_w}{M_n}$$

where W_i is the weight of each fraction (g), N_x is the number of polymer molecules, M_w is the weight-average molecular weight, and M_n is the number-average molecular weight.

PSA tape preparation

Emulsion PSAs mixed with a 30 wt % aqueous tackifier dispersion based on a phenolic acid derivative, E-100, as a tackifier, obtained from Nitto Denko Co., Ltd. (Japan), were coated with stainless steel (SS) applicator (model SA-201, Tester Sangyo) directly onto a 38- μm corona PET film provided by Nitto Denko to give a 50- μm -dry-thickness PSA tape. After the PSA tape was dried in an oven at 130°C for 3 min, it was cooled at room temperature. PSA tests were performed at room temperature, and the samples were acclimatized at room temperature for 24 h before the measurements.

TABLE I
Miscibility of the Monomer Emulsion and PBA Latex in the Presence of Toluene or EA

Organic solvent	Amount (wt %)	Appearance	
		Monomer emulsion	PBA latex
Toluene	5	Miscible	Phase separation
	10	Phase separation	Phase separation
	20	Phase separation	Phase separation
	30	Phase separation	Phase separation
EA	5	Miscible	Miscible
	10	Miscible	Miscible
	15	Miscible	Miscible
	20	Slight phase separation	Phase separation
	30	Phase separation	Phase separation

The prepared PSA tapes were examined for surface homogeneity with a Krüss model G10 contact-angle machine (Hamburg, Germany). The PSA tape was placed on the glass plate, and then the deionized water was diminutively dropped onto this sample. After a dwelling time of 2 min, the contact angles between the water drop and PSA sample on the left- and right-hand sides were recorded. Ten measurements were taken to get an average value.

PSA testing

PSA testing was performed in accordance with JIS Z 0237, ASTM D 3330, and ASTM D 3121 and could be divided into three main categories: the adhesion test, shear holding power test, and rolling ball tack test.

Analysis of the adhesion properties

The PSA tape (20 × 130 mm²) was applied to SS as a substrate with a 2-kg SS roller to make good contact. After a dwelling time of 10 min, the 180° peel off the substrate was clamped to the grips of the tensile tester

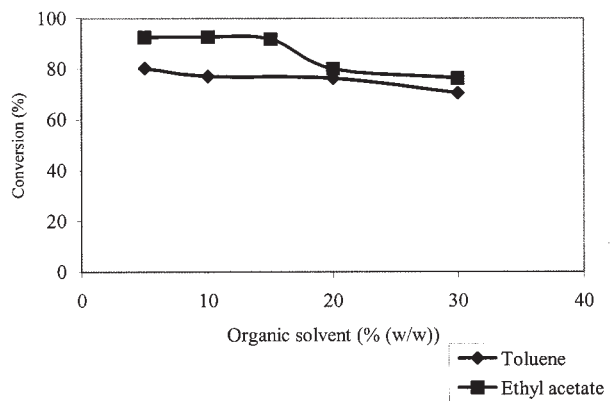


Figure 2 PBA conversion with emulsion polymerization in the presence of an organic solvent.

TABLE II
Particle Size and M_v of the PBA Latex with EA

Organic solvent	Amount (wt %)	Particle size (μm)	M_v ($\times 10^5$)
—	—	0.088	10.0 \pm 0.2
EA	5	0.150	10.1 \pm 0.2
	10	0.150	10.3 \pm 0.2
	15	0.150	10.7 \pm 0.2
	20	0.150	17.0 \pm 0.2

machine (model 5569, Instron, Norwood, MA). The upper grip was brought upward at a crosshead speed of 150 mm/min until detachment occurred. The peel force at a tape distance of 25–75 mm was recorded in terms of N/100 mm and the maximum force.

Analysis of the shear holding power properties

A 25 × 25 mm² strip was cut from the PSA tape. The strip was laminated against SS with a 2-kg SS roller to make contact areas of 25 × 25 mm². After a dwelling time of 10 min, the sample was clamped to the grip of a tester with a 550-g load at 40°C. The shear holding power was measured by the counting of the time that elapsed between the application of the load and the completed separation of the tape from SS.

Analysis of the tack properties

The tack properties were determined with a rolling ball tester. Steel balls of different sizes were released from the top of an incline with an angle of 30°, were allowed to accelerate down, and were rolled onto adhesive tape. The tack was obtained by the measurement of the distance that the ball traveled across the adhesive before stopping, and the maximum ball size was recorded.

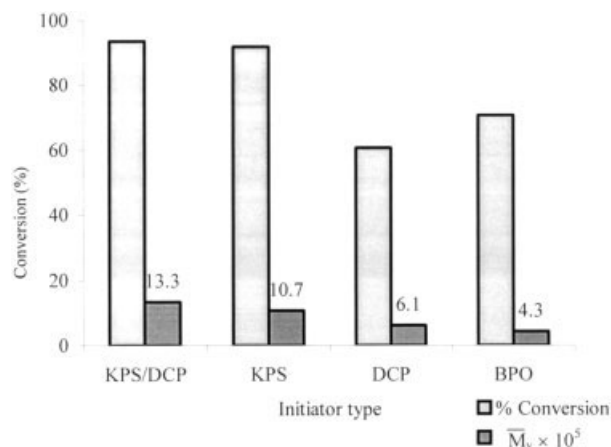


Figure 3 Effect of the initiator type on the monomer conversion and M_v .

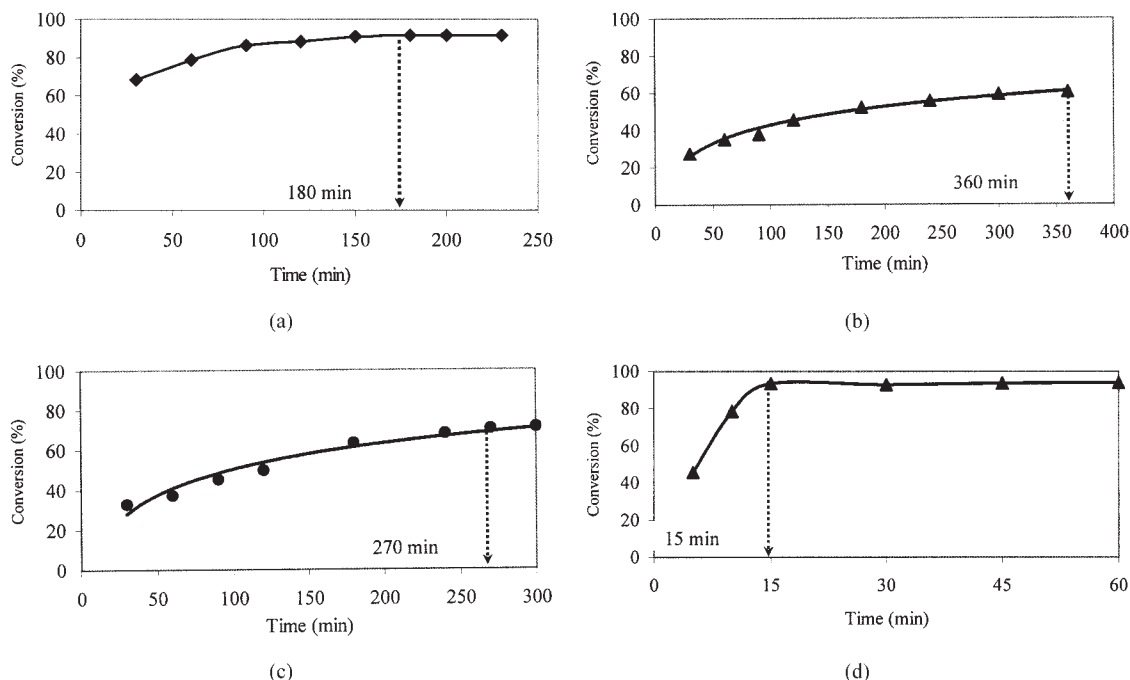


Figure 4 Relationship between the conversion and polymerization time of BA in the presence of EA with the initiators (a) KPS, (b) DCP, (c) BPO, and (d) KPS/DCP.

RESULTS AND DISCUSSION

Emulsion polymerization of *n*-BA in the presence of an organic solvent

Effects of the types and amounts of the organic solvent

Toluene and EA were used as organic solvents to mix into the polymerization of *n*-BA. The compatibility of the obtained mixture, that is, the monomer emulsion, was reported as an occurrence of phase separation or good miscibility. Table I shows the miscibility of toluene or EA with the monomer emulsion and PBA latex, which were identified by visual testing after the mixture was left for 15 min. The results indicated that in the case of BA with toluene, the monomer emulsions changed from good miscibility to phase separation with the toluene concentration higher than 10 wt %. On the other hand, in the case of *n*-BA with EA, complete phase separation was observed only when the concentration of EA was higher than 30 wt %. It could be deduced that the solubility parameter and type of hydrogen bonding of EA were closer to those of the *n*-BA monomer than those of toluene. That is, the solubility parameters of *n*-BA, toluene, and EA were reported to be 8.8, 8.9, and 9.1, respectively.⁸ Nevertheless, the hydrogen bonding of toluene was poor, whereas that of *n*-BA and EA was moderate. Hence, it was observed that when the maximum concentration of EA, 15 wt %, was mixed with BA, good miscibility was shown for the monomer emulsion. Thus, it could be deduced that EA showed better

miscibility with BA than toluene. Figure 2 shows clearly that the conversion was affected by the amount of the organic solvent in the reaction mixture. As mentioned previously, it implies that EA (5–15 wt %) could be miscible with *n*-BA, and it gave a higher conversion than toluene. However, the conversion decreased with an increasing concentration of EA (20 and 30 wt %). This might be due to the occurrence of phase separation, as described previously. Table II indicates that the PBA latex with EA under all conditions had a slightly larger particle size than PBA without EA (0.088 μm). The M_w value was very high for all conditions and showed the highest value at 20 wt % EA. This might be due to the swelling of PBA particles by EA, which resulted in the fusion of particles to form bigger particles. This effect was expected to give good

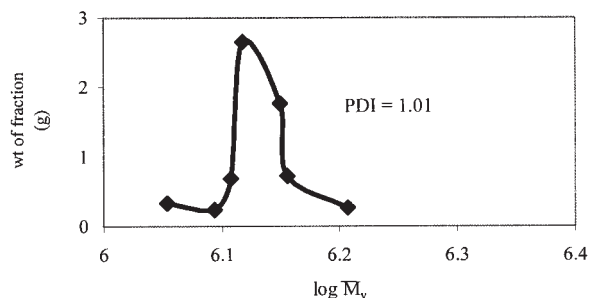


Figure 5 MWD of PBA polymerized with the KPS/DCP combination initiator (0.02/0.008 w/w), as determined by solvent fractionation (PDI = polydispersity index).

properties for the PSA tape made from this PBA emulsion, as discussed in a subsequent section.

Effect of the initiator

n-BA homopolymerization in the presence of 15 wt % EA was carried out with different initiators, that is, KPS, BPO, DCP, and a combination of KPS and DCP. Figure 3 shows that the combination of KPS and DCP in ratios of 0.02 and 0.008 wt % gave a conversion close to that of KPS alone. On the other hand, the use of only DCP or BPO as the initiator gave the lower conversion. This might be due to the half-life of each initiator. That is, the half-lives of DCP and BPO are approximately 13 and 7.3 h at 115 and 70°C, respectively.⁸ It is remarkable that M_v of PBA with a combined initiator system was much higher than that obtained from a single initiator system. This might be due to the efficiency of each initiator as well as the polymerization temperature. Figure 4(a–d) shows the relationship between the conversion and polymerization time of PBA with various types of initiators (KPS, DCP, BPO, and a combination of KPS and DCP, respectively). The polymerization times of 180, 360, 270, and 15 min were enough to get the maximum conversion for KPS, DCP, BPO, and a combination of KPS and DCP, respectively. The KPS/DCP initiator system showed the highest rate of polymerization; almost 100% conversion was obtained within 15 min. Figure 5 shows the MWD of PBA prepared with the KPS/DCP combination initiator obtained with the solvent fractionation technique. Figure 6 shows the PSD of the same sample observed with a particle size analyzer. Both distribution curves are very narrow. Table III shows the polydispersity index of the samples prepared with various ratios of KPS to DCP. The polydispersity index was very close to that for the monodisperse case. These findings strongly

TABLE III
Polydispersity Index of PBA Polymerized by the KPS/DCP Combination Initiator

KPS/DCP ratio (w/w)	Polydispersity index
0.02/0.02	1.03
0.02/0.008	1.02
0.02/0.006	1.02
0.02/0.004	1.01

support the idea that a combination of KPS and DCP as an initiator could give a homogeneous particle size derived in a very short polymerization time.

Analysis of the PSA properties

After the polymerization was complete, the latex was applied to the preparation of a PSA tape having a 50- μ m dry thickness with a PET film as an adherent and SS as an applicator. The latex, including a suitable amount of the solvent, could be cast more easily than the latex without the addition of the solvent. This might be due to the effect of the solvent, which swelled the latex particles and increased the interaction among the particles, leading to a high viscosity of the resulting latex.

Analysis of the homogeneity of the prepared PSA tape by the contact angle

The homogeneity thickness of the PSA tapes cast from various kinds of PBA latices was evaluated by the measurement of the contact angle against distilled water at 25°C with the sessile drop equilibrium contact-angle method. Table IV indicates that almost all the PBA samples showed a deviation of the contact-

TABLE IV
Contact Angles of Various PSA Tapes Prepared by Different Recipes

Sample	Contact angle (°)	Sample	Contact angle (°)
30wt % <i>n</i> -BA	95.7 ± 0.8	<i>n</i> -BA + KPS/DCP (0.02/0.004% w/w) + 15wt % EA	96.7 ± 0.8
35wt % <i>n</i> -BA	96.7 ± 1.8	<i>n</i> -BA + KPS/DCP (0.02/0.002% w/w) + 15wt% EA	96.6 ± 2.9
40wt% <i>n</i> -BA	96.4 ± 1.2	<i>n</i> -BA + AA + KPS + 15wt% EA	95.9 ± 1.7
50wt% <i>n</i> -BA	97.1 ± 1.3	<i>n</i> -BA + AA + KPS/DCP (0.02/0.008% w/w) + 15wt% EA	96.2 ± 0.8
60wt% <i>n</i> -BA	96.8 ± 1.0	PBA + 10wt % EA	96.2 ± 0.5
<i>n</i> -BA + KPS + 15wt % EA	96.2 ± 1.0	PBA + 20wt % EA	97.1 ± 1.3
<i>n</i> -BA + KPS + 5wt % TL	96.8 ± 2.6	PBA + 30wt % EA	96.2 ± 1.6
<i>n</i> -BA + KPS/DCP (0.02/0.02% w/w) + 15wt % EA	96.9 ± 1.1	PBA + 40wt % EA	96.4 ± 2.6
<i>n</i> -BA + KPS/DCP (0.02/0.008% w/w) + 15wt % EA	96.5 ± 0.4	PBA + 50wt % EA	96.8 ± 1.0
<i>n</i> -BA + KPS/DCP (0.02/0.006% w/w) + 15wt % EA	96.4 ± 1.6	PBA + 60wt % EA	97.2 ± 2.1

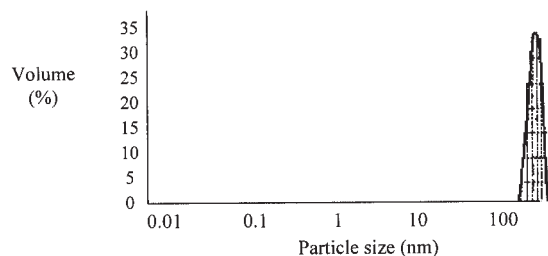


Figure 6 PSD of PBA synthesized with the KPS/DCP combination initiator (0.02/0.008 w/w).

angle value in the range of about 2° . However, some samples showed a deviation angle value than 2° , which indicated the presence of surface roughness on the surface of the PSA tapes.⁹ This might be due to the sample being cut from the edge of the PSA specimen. It is possible that the thickness at the edge of the PSA specimen was not as uniform as at the center. This suggests that an appropriate representative sample for contact-angle testing should be a sample from the center of the PSA specimen.

Analysis of the adhesive properties

The prepared PSA tapes were used to investigate the 180° peel force with an Instron tensile tester at a speed rate of 150 mm/min. In the course of the polymerization of *n*-BA, 15 wt % EA was added to the latex to compare the peel force of adding EA between prepolymerization and postpolymerization. In the case of adding EA at the postpolymerization, as shown in Figure 7, as the amount of EA increased, the average peel force slightly increased from 2.48 N/100 mm to the maximum value, 4.30 N/100 mm, which was observed at 30 wt % EA; after that, the average peel force slightly decreased from 3.91 to 3.62 N/100 mm. How-

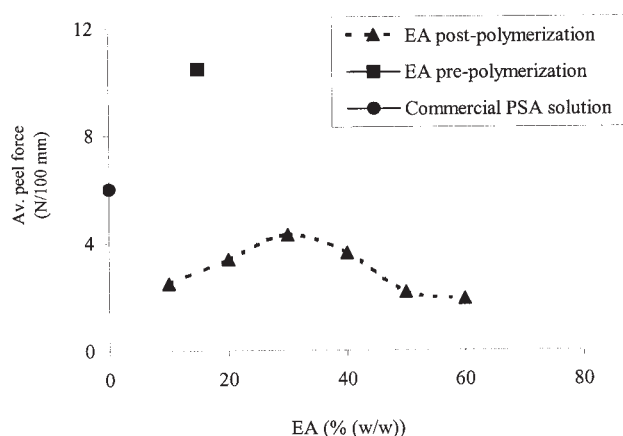


Figure 7 Comparison of the average peel force of PBA in the presence of EA between prepolymerization and postpolymerization.

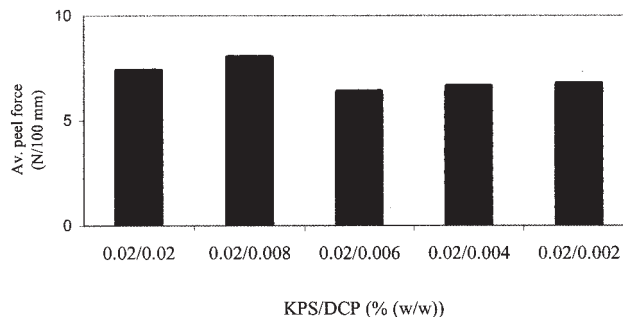


Figure 8 Average 180° peel force of PBA with various combinations of KPS and DCP.

ever, all cases of postpolymerization showed a lower average peel force than that of 15 wt % EA addition at prepolymerization, which was 10.46 N/100 mm. It might be explained that in the case of adding EA at prepolymerization, EA went into the polymer particles and swelled PBA. Eventually, it might have assisted in combining the polymer particles. Thus, the average peel force of PBA with EA at prepolymerization was higher than that of PBA, which was only 5.99 N/100 mm. In the case of postpolymerization, EA could not miscible with PBA particles as in the case of prepolymerization. Furthermore, when EA was added after the completion of postpolymerization, phase separation slightly occurred and raised the coagulum content. This might be due to the amounts of the surfactants; that is, the effectiveness of the surfactants was not used to protect the particles as the polymerization proceeded. The average peel adhesion of the PBA homopolymer was investigated with the variation of the ratio of the KPS/DCP combination initiator, as shown in Figure 8. PSA tapes, which were produced from PBA polymerized by various DCP and BPO ratios, could not be subjected to measurements for the 180° peel force because the tapes were slippery with SS as the substrate. The wettability between the PSA tapes and the substrate seemed to be inferior. This figure implies that every ratio of KPS to DCP showed almost the same average peel value between 6.34 and 7.36 N/100 mm. This might be due to the homogeneity of the PBA latex in these cases.

Analysis of the shear holding power properties

Figure 9 shows the contact time against the amount of EA in the case of the addition of EA at postpolymerization, that is, 10–60 wt %. The contact time decreased as the concentration of EA increased until the maximum concentration of EA reached 20 wt %. Then, the contact time was kept constant as long as 1 day, whereas the concentration of EA added to the latex was greater than 20 wt %. This implies that with the

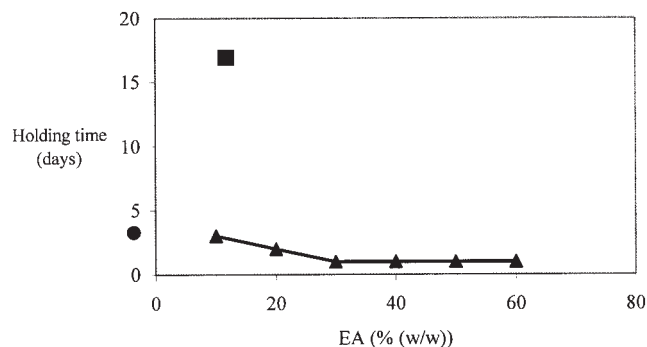


Figure 9 Plot of the contact time versus the amount of EA added to the latex at postpolymerization.

addition of EA to the latex at postpolymerization, EA was compatible with PBA to a limited extent.

Analysis of the tack properties

The rolling ball test was conducted with a 30° inclined plate according to JIS Z 0237 and ASTM D 3121. Table V shows the tack testing results; the ball, which could be stuck to the PSA tape, had a diameter of 5 or 10 mm or was the maximum size,. A greater ball distance and a low maximum ball diameter meant lower tackiness. The tack results showed a similar trend for the PBA homopolymer with KPS and KPS/DCP combination initiators and the addition of EA for prepolymerization. With ball diameters of 5 and 10 mm, the distances were 5 mm and 10–15 mm, respectively. Thus, it could be inferred that the two ball diameters showed close values. Moreover, the maximum ball sizes were 15–18 mm, and this showed the closeness of the ball dis-

tance. This implied that PBA produced with the KPS and KPS/DCP combination initiator systems gave similar tack properties. As mentioned previously, it could be inferred that all the PSA samples with EA at prepolymerization had higher tackiness than those produced by the addition of EA at postpolymerization.

CONCLUSIONS

PSA tapes made from PBA latex prepared by emulsion polymerization have not been commercially used because of the poorer adhesion properties with respect to PSA made from PBA by solution polymerization. This work was an attempt to elucidate a new emulsion polymerization technique for producing polyacrylate latices for PSAs having excellent adhesion properties. A novel emulsion polymerization was performed through the addition of an organic solvent such as EA to the monomer to make swelled polymer particles, and we expected the latex to improve the properties of PSA by the fusion of emulsion polymer particles. The high-performance PBA latex could be synthesized by the emulsion polymerization of 50 wt % *n*-BA mixed with 15 wt % EA with a new initiator system composed of KPS and DCP. The initiator system gave high-molecular-weight PBA with a polydispersity index of 1.01 and a particle size of 0.150 μm in 15 min of polymerization at 70°C. The PSA tape produced from the resulting prepolymerized PBA latex showed adhesion properties better than those obtained by the postpolymerization and conventional solution polymerization.

TABLE V
Tack Properties of Various PSA Tapes Prepared by Different Recipes

Sample	Diameter of the maximum ball (mm)	Ball distance		
		Maximum	$\phi = 5$ mm	$\phi = 10$ mm
<i>n</i> -BA + KPS + 15wt % EA	15	20 \pm 5	5 \pm 0	15 \pm 5
<i>n</i> -BA + KPS/DCP (0.02/0.02% w/w) + 15wt % EA	18	20 \pm 5	10 \pm 5	15 \pm 5
<i>n</i> -BA + KPS/DCP (0.02/0.008% w/w) + 15wt % EA	15	20 \pm 5	5 \pm 0	10 \pm 5
<i>n</i> -BA + KPS/DCP (0.02/0.006% w/w) + 15wt % EA	15	25 \pm 5	5 \pm 0	15 \pm 5
<i>n</i> -BA + KPS/DCP (0.02/0.004% w/w) + 15wt % EA	16	20 \pm 5	5 \pm 0	15 \pm 5
<i>n</i> -BA + KPS/DCP (0.02/0.002% w/w) + 15wt % EA	15	20 \pm 5	5 \pm 0	10 \pm 5
PBA + 10wt % EA	3	20 \pm 5	—	—
PBA + 20wt % EA	2	10 \pm 5	—	—
PBA + 30wt % EA	2	15 \pm 5	—	—
PBA + 40wt % EA	2	15 \pm 5	—	—
PBA + 50wt % EA	2	15 \pm 5	—	—
PBA + 60wt % EA	2	15 \pm 5	—	—

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