

# Effect of Network Morphology on Adhesive Performance in Emulsion Blends of Acrylic Pressure Sensitive Adhesives

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**ABSTRACT:** High-gel containing latices and gel-free latex were blended at various weight ratios. The high-gel containing latices was made of poly(2-ethyl hexylacrylate-*stat*-acrylic acid) and the gel-free latex was made of poly(2-ethyl hexylacrylate-*stat*-acrylic acid-*stat*-isobutoxymethyl acrylamide) using semicontinuous emulsion polymerization. Films were cast at room temperature and dried at 121°C for 10 min. Adhesive performance was evaluated in terms of loop tack, peel, and shear holding power. It was found that interlinking the microgels by the linear polymer due to the isobutoxymethyl acrylamide-acrylic acid reaction in the film when heated gave synergistic effects in increasing shear. This interlinking could take place only if the molecular weight between crosslinks ( $M_c$ ) of the microgels was greater than the entanglement molecular weight of the linear polymer ( $M_e$ ), and if the weight average molecular weight of the linear polymer ( $M_w$ ) was greater than  $2 \times M_e$ . © 2001 John Wiley & Sons, Inc. *J Appl Polym Sci* 81: 2109–2117, 2001

**Key words:** emulsion blends; PSA; acrylic; microgel

## INTRODUCTION

The subject of polymer blends has been studied rigorously for the past three decades.<sup>1,2</sup> Despite the extensive literature covering polymer blends prepared by melt and solution mixing, blends prepared by mixing two or more latices are much less known.<sup>3</sup> In melt and solution blends, two high molecular weight polymers with significantly different solubility parameters and no specific interaction will result in a two layer film when cast. On the other hand, an emulsion blend can provide discrete film morphology if the minor component

does not self-coalesce. This condition depends on the emulsion stability,  $T_g$ , and molecular weight of the minor component. An example of the utility of this concept is the emulsion blend of silicone and acrylic pressure sensitive adhesives (PSAs).<sup>4</sup> By having silicone PSA as the dispersed phase, adhesion to a low energy substrate such as silicone coated paper was significantly increased. Other examples are the emulsion blend of acrylic acid (AA) containing acrylic PSA, and acrylamide (AM) containing acrylic PSA.<sup>5</sup> Intermolecular interaction between AA and AM in the cast film reduced the peel strength of a substrate after the bond had been aged.

Repulvable or water-dispersible PSA can be prepared by blending high molecular weight acrylic and low molecular weight-high acid con-

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tent acrylic latices.<sup>6</sup> The low molecular weight-high acid content acrylic polymer acts as tackifier and aids in water-dispersibility at the same time. Another way to prepare water-dispersible PSA is by blending acrylic and poly(alkoxyalkyl) acrylate latices.<sup>7</sup>

Interpenetrating elastomeric network (IEN) can be prepared by blending linear poly(urethane-urea) and poly(butadiene-acrylonitrile) latices and curing them *in situ* after the film has been cast.<sup>8</sup> Partial interpenetration of the two networks takes place during the interdiffusion stage of film formation. Gross phase separation is prevented by the formation of the networks *in situ*. The IEN film has higher tensile strength than either one of the constituents.

In emulsion blends, thermodynamic compatibility does not necessarily result in mechanical compatibility or synergism.<sup>3</sup> Film cast at room temperature from emulsion blends of poly(vinyl acetate) and poly(vinyl acetate-*stat*-ethylene) shows low elongation at break.<sup>3</sup> However, when the blend is melt mixed and compression molded, it shows much higher elongation at break. Compliance mismatch due to the large difference in  $T_g$  between the two polymer latices has been implicated as the cause of the low elongation at break in room temperature cast film. In emulsion blend of high  $T_g$  rosin ester tackifier and acrylic PSA, the lack of entanglement between the linear polymer and the microgels causes a significant drop in shear holding power of the PSA film.<sup>9</sup> This can be avoided if the linear polymer has weight-average molecular weight ( $M_w$ ) of at least twice the entanglement molecular weight ( $M_e$ ), and the microgels are chemically linked with the linear polymer in the film.<sup>10</sup> The linkages are created from the reaction of isobutoxymethyl acrylamide (IBMA)-containing acrylic PSA in the film when heated. Microgels are a product of chain

transfer to polymer reaction during semicontinuous emulsion polymerization of acrylic PSA.<sup>11</sup>

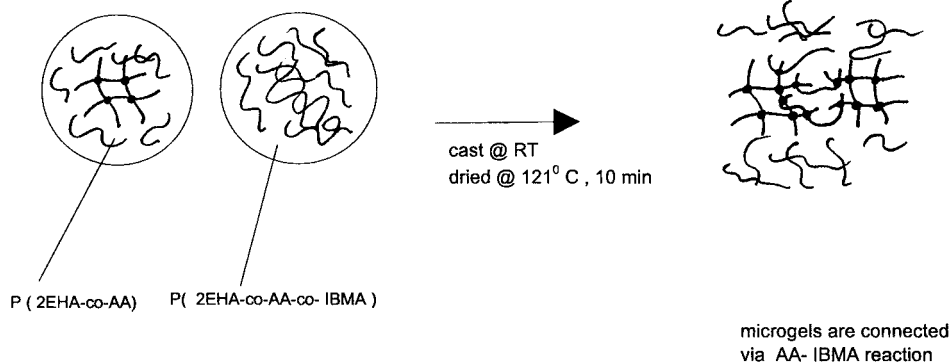
This article deals with blends of high-gel containing poly(2-ethyl hexylacrylate-*stat*-acrylic acid) and essentially gel-free poly(2-ethyl hexylacrylate-*stat*-acrylic acid-*stat*-IBMA) latices. Poly(2-ethyl hexylacrylate) is the lowest  $T_g$  acrylic PSA, and it has excellent low temperature adhesion.<sup>12</sup> Unfortunately, because of its high  $M_e$  ( $M_e \sim 40,000$  g/mol), this polymer provides very low shear holding power,<sup>10</sup> and therefore it may present a problem in PSA label converting and high temperature printing.

The goal of this research was to understand the effect of crosslink density and reactivity of the microgels on forming interlinkages with the linear polymer in the film when heated. Such interlinkages would bring a synergistic effect by increasing the shear strength of the PSA film. The interlinkages were provided by the reaction of acrylic acid in the microgels and IBMA in the linear polymer when the film was heated, as shown in Figure 1. The blend composition having the highest synergistic effect was compared with solvent borne acrylic in terms of loop tack, peel, and shear for both neat and tackified compositions. In addition, the results were also compared with a single emulsion system studied earlier.<sup>10</sup>

## EXPERIMENTAL

### Materials

The 2-ethyl hexylacrylate (2EHA) and AA monomers were all commercial grades from Elf-Atochem. IBMA was obtained from CYTEC. Allyl methacrylate (AMA) and poly(ethylene glycol-diacrylate) (PEG500-diacrylate) were obtained from



**Figure 1** Interlinking of the microgels by the linear polymer.

Aldrich Chemicals. Chain transfer agent n-dodecyl mercaptan (n-DDM) was obtained from Aldrich.

Water soluble AIBN initiator, Wako V-50® [2,2'-azobis (2-amidino propane) dihydrochloride], was obtained from Wako Chemicals USA, Inc. Ammonium persulfate (APS) initiator was obtained from FMC Corporation. Oil soluble AIBN, Vazo® 67, was obtained from DuPont.

The surfactant used was nonyl-phenol ethoxylate sulfate, having four ethylene-oxide units (Polystep® B-27) from Stepan Chemical Company. Its activity in H<sub>2</sub>O is 30% by weight.

The aqueous tackifier dispersion was based on glycerol ester abietic acid ( $M_w = 940$ , PDI = 1.1, DMA  $T_g = 64^\circ\text{C}$ ), Snowtack® 920 from AKZO NOBEL. The mean particle diameter was  $514 \pm 202$  nm and the solid content was 58%. The dried version of this tackifier (including the surfactant) was dissolved in toluene to be used in tackification of solvent borne acrylic PSA.

The NH<sub>4</sub>OH (25% concentration by weight in H<sub>2</sub>O) was obtained from Textile Chemicals. Kathon® LX (biocide) was obtained from Rohm and Haas.

#### Preparation of Acrylic PSA Using Semicontinuous Emulsion Polymerization

The latices were prepared by semicontinuous emulsion polymerization using V-50® initiator at 60°C. The detailed procedure has been described elsewhere.<sup>10</sup> The mean particle diameters were  $245 \pm 30$  nm as determined by dynamic light-scattering.

#### Preparation of Acrylic PSA Using Semicontinuous Solution Polymerization

The acrylic solution PSA was prepared by semicontinuous solution polymerization using Vazo® 67 initiator at 73°C. The detailed procedure has been described elsewhere.<sup>10</sup> The molecular weight averages,  $M_w$  and  $M_n$ , of the P(2EHA-*stat*-AA) 97.5/2.5 by weight were 259,000 and 49,400 g/mol, respectively, prior to crosslinking with Al acetyl acetate (AAA).

#### PSA Testing

The preparation of emulsion and solvent borne cast films has been described elsewhere.<sup>10</sup> A standard drying temperature of 121°C for 10 min was used to dry the films.

PSA testing was done at 23°C and 50% R.H. and the samples were climatized into this condition 24 h before testing.

Loop tack and 90° peel were done off stainless steel and HDPE substrates as described elsewhere.<sup>10</sup> Shear holding power was done off stainless steel using a  $1.27 \times 1.27$  cm PET coated strip and 4.9 N hanging weight, and the time to failure was recorded.

#### Solvent Extraction and Swelling

Determination of gel content and swelling was carried out using a membrane gel partitioning method, the detailed procedure of which has been described elsewhere.<sup>10</sup> For the gel swell experiment, 5 μm pore size PTFE and 0.22 μm PVF<sub>2</sub> membranes were used concurrently to confirm the results. The molecular weight between crosslink points ( $M_c$ ) was calculated from the swelling data.<sup>10</sup>

#### Molecular Weight Characterization

The THF soluble fraction from the adhesive extraction experiment above was injected into the GPC column (Water Alliance 2690), the detailed procedure of which has been described elsewhere.<sup>10</sup>

#### Determination of $M_e$

The  $M_e$  of polydisperse emulsion and solution polymers was calculated from the plasticizer model described elsewhere<sup>10</sup> given the  $M_e$  of monodisperse P(2EHA) to be 37,432 g/mol.

## RESULTS AND DISCUSSION

#### Effect of $M_c$ on Adhesive Performance

The molecular properties of the latices were measured on room temperature (RT) cast and dried samples in order to assess them prior to application of heat. The properties are shown in Table I.

Emulsion 1 was essentially a gel-free latex made by adding 0.1 wt % n-DDM into the 2EHA/AA/IBMA monomer mixture. This latex was blended with one of the other latices, i.e., emulsions 2–6, which contained a high level of microgels at 100/0, 75/25, 50/50, 25/75, and 0/100 weight ratios of emulsion 1/emulsions 2–6.

Emulsion 2 was a high-gel containing latex made by adding 0.5 wt % AMA into 2EHA/AA monomer mixture. Emulsion 2 had  $M_c \ll M_e$ , i.e., a very tight network.

Emulsion 3 was a high-gel containing latex made by copolymerizing a 2EHA/AA monomer

**Table I Molecular Properties of Emulsion Films Cast and Dried at Room Temperature**

Emulsion <sup>a</sup>	Gel Content (%)	$M_c$ (g/mol)	$M_e$ (g/mol) <sup>b</sup>	$M_w$ (g/mol)	$M_n$ (g/mol)
1	3	—	50 K	210 K	50 K
2	94	17 K	154 K	80 K	18 K
3	70	110 K	47 K	234 K	28 K
4	83	60 K	97 K	143 K	20 K
5	80	110 K	42 K	269 K	36 K
6	76	90 K	42 K	228 K	54 K

(1) 2EHA/AA/n-DDM/IBMA 96.9/2.5/0.1/0.5 by weight.

(2) 2EHA/AA/AMA 97/2.5/0.5.

(3) 2EHA/AA 97.5/2.5.

(4) 2EHA/AA/PEG500-diacrylate 95.5/2.5/2.

(5) 2EHA/AA 97.5/2.5 with 0.112 pphm APS as chaser.

(6) 2EHA/AA 96/4.

<sup>a</sup> Latices were prepared by semicontinuous emulsion polymerization at 60°C using V-50® initiator. Particle size was 245 ± 30 nm.<sup>b</sup> Calculated from the plasticizer model.

mixture. The microgels were a product of chain transfer to polymer reaction during semicontinuous emulsion polymerization.<sup>11</sup> Emulsion 3 had  $M_c > M_e$ , i.e., a loose network.

Emulsion 4 was made by copolymerizing 2 wt % PEG500-diacrylate into 2EHA/AA monomer mixture. The level of crosslinking macromonomer used here was equimolar to the AMA used in emulsion 2. The  $M_e$  of emulsion 1/emulsion 4 blends ranged from 52,000 to 66,000 g/mol at 75/25 to 25/75 weight ratios. Therefore, these blends had  $M_c \approx M_e$  because the  $M_c$  of emulsion 4 was 60,000 g/mol, as shown in Table I.

Emulsion 5 was made by adding 0.112 parts per hundred of monomer (pphm) APS late in the polymerization P(2EHA-*stat*-AA) latex. This was aimed to increase the reactivity of the microgel with the IBMA from the linear polymer (emulsion 1) in the film when heated. It was known that IBMA reactivity could be increased with increas-

ing acidity.<sup>13</sup> Persulfate is more acidic than V-50®.

Emulsion 6 was made by adding 4 wt % AA as comonomer mixture, instead of 2.5 wt %. Again, the aim was to increase the reactivity of the microgel with the IBMA from the linear polymer in the film when heated. Both emulsion 5 and 6 had  $M_c > M_e$ .

Table II shows the adhesive performance of emulsion 1/emulsion 2 blends at 100/0, 75/25, 50/50, 25/75, and 0/100 weight ratios. The films were cast at RT and dried at 121°C for 10 min. As expected, increasing the level of emulsion 2 increased the gel content, and consequently the shear strength was increased at the expense of lowering peel and tack. The results were further clarified by plotting loop tack, peel, and shear off stainless steel versus gel content, as shown in Figure 2. Because emulsion 1 was essentially gel-free at RT, its network morphology after drying

**Table II Effect of Molecular Weight between Crosslink Points ( $M_c$ ) on Adhesive Performance**

$M_c < M_e$					
Em. 1/Em. 2	Gel Content (%)	L. Tack SS (N/m)	L. Tack HDPE (N/m)	90° Peel SS (N/m)	90° Peel HDPE (N/m)
100/0	20	667	298	614	228
75/25	33	561	263	544	210
50/50	50	333	228	526	70
25/75	70	281	105	175	53
0/100	90	123	88	70	35

PSAs were coated on 50 μm PET at 30 μm dry film thickness and dried at 121°C for 10 min.

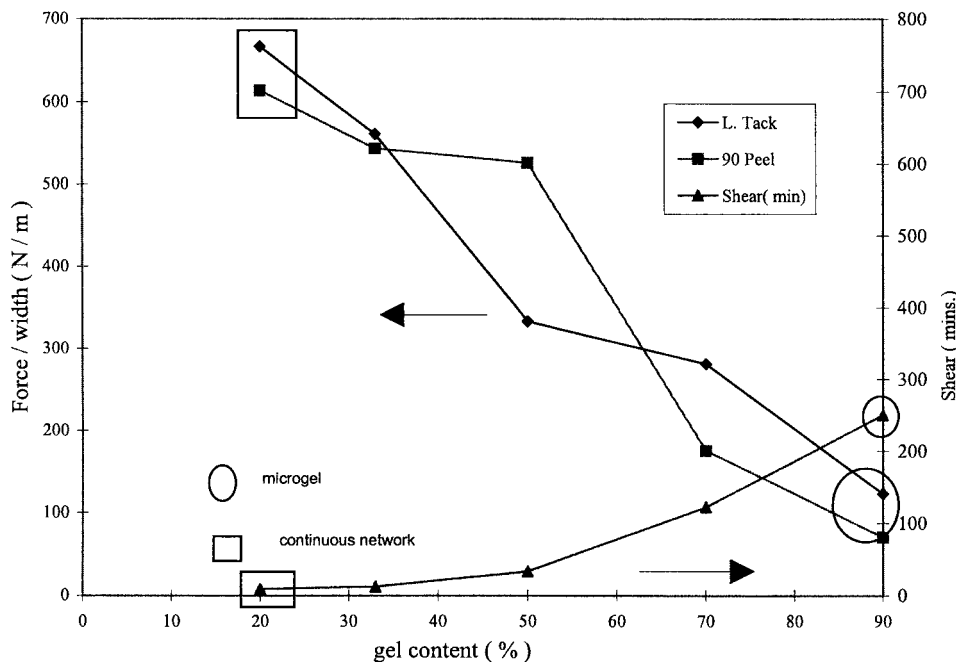


Figure 2 Adhesive performance of emulsion 1/emulsion 2 blends ( $M_c < M_e$ ).

the film at 121°C for 10 min would be continuous. In contrast, emulsion 2 contained a high level of microgels, which retained their morphology in the film. Emulsion 1/emulsion 2 blends showed no synergistic effects at any compositions.

#### Effect of Tube Diameter

As expected, because  $M_c < M_e$ , the linear polymer was not able to entangle with the microgels, and therefore little reaction between IBMA and AA took place. Zosel and Ley reported that crosslinked poly(butyl methacrylate) latex film with  $M_c < M_e$  had fracture toughness 130 times lower than that with  $M_c > M_e$ .<sup>14</sup> The ability of the microgels to entangle with the linear polymer is required before the reaction can take place.

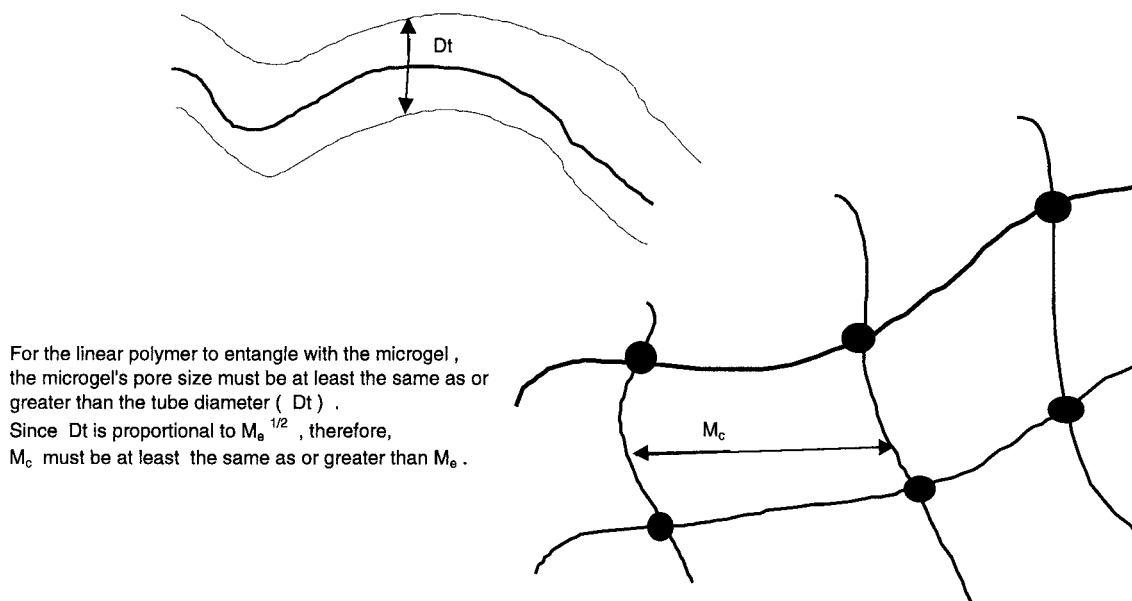
According to the reptation model, the tube diameter of a linear polymer is equal to  $M_e^{1/2}$  times the constant ratio  $(\langle R^2 \rangle_0 / M)^{1/2}$ ,<sup>15</sup> which equals about 0.7 for many acrylic polymers. For poly(2-ethyl hexylacrylate), such as emulsion 1, the tube diameter is approximately 70 Å. In order for the linear polymer to penetrate into the microgels, the chain dimension of each segment of the network must be at least 70 Å. This would correspond to an  $M_c$  value of approximately 50,000 g/mol, i.e.,  $\approx M_e$  of the linear polymer. This is a lower bound approximation that assumes no competing reactions, as discussed below. Figure 3 shows the schematic morphology of the microgel's interpenetration by the linear polymer.

#### Effect of Gel Content

Figure 4 shows loop tack strength, peel resistance, and shear strength off stainless steel of emulsion 1/emulsion 4 blend films plotted versus the gel content. The films were cast and dried with the same conditions as above. It is interesting to observe that no synergistic effect was observed in these blends, although  $M_c \approx M_e$ . In order to understand the reason for this lack of synergism, one needs to examine the other two competing reactions besides the one shown in Figure 1. In addition to the reaction between IBMA from the linear polymer and AA from the microgels, IBMA from the linear polymer could self-crosslink and it could also crosslink with the AA from the sol fraction of the high-gel containing emulsion 4. The linear polymer had an average of four entanglements per molecule ( $M_w/M_e \approx 4$ ). The microgels had taken the length equivalent to one entanglement away from the linear polymer because its  $M_c \approx M_e$ . Therefore, the linear polymer still had the length equivalent to three entanglements, which upon heating would favor self-crosslinking and the formation of a phantom network, i.e., a separate network that did not link the microgels.

Figure 5 shows loop tack, peel, and shear off stainless steel of emulsion 1/emulsion 3 blend films plotted versus the gel content. The films were cast and dried with the same conditions as above. With  $M_c > M_e$ , precisely  $M_c \approx 2M_e$ , the





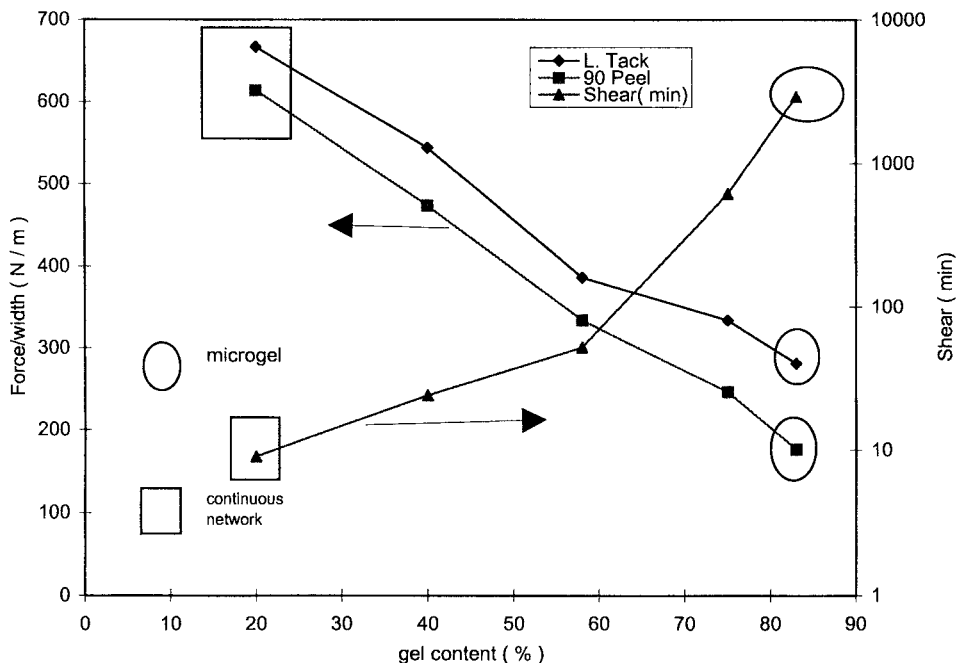
**Figure 3** Interpenetration of the microgel by the linear polymer.

microgels started to react with the linear polymer and mild synergism in shear holding power was obtained, as shown in Figure 5. The blends at 50/50 and 25/75 emulsion 1/emulsion 3 weight ratios had a higher shear holding power than either one of the constituents. In essence, with  $M_c/M_e \approx 2$  and  $M_w/M_e \approx 2$ , the microgels had the same probability of reaction with the linear

polymer as the other two competing reactions mentioned above.

**Effect of Microgel Reactivity on Adhesive Performance**

Figure 6 shows adhesive performance of emulsion 1/emulsion 5 blend films at the same weight ra-



**Figure 4** Adhesive performance of emulsion 1/emulsion 4 blends ( $M_c \approx M_e$ ).

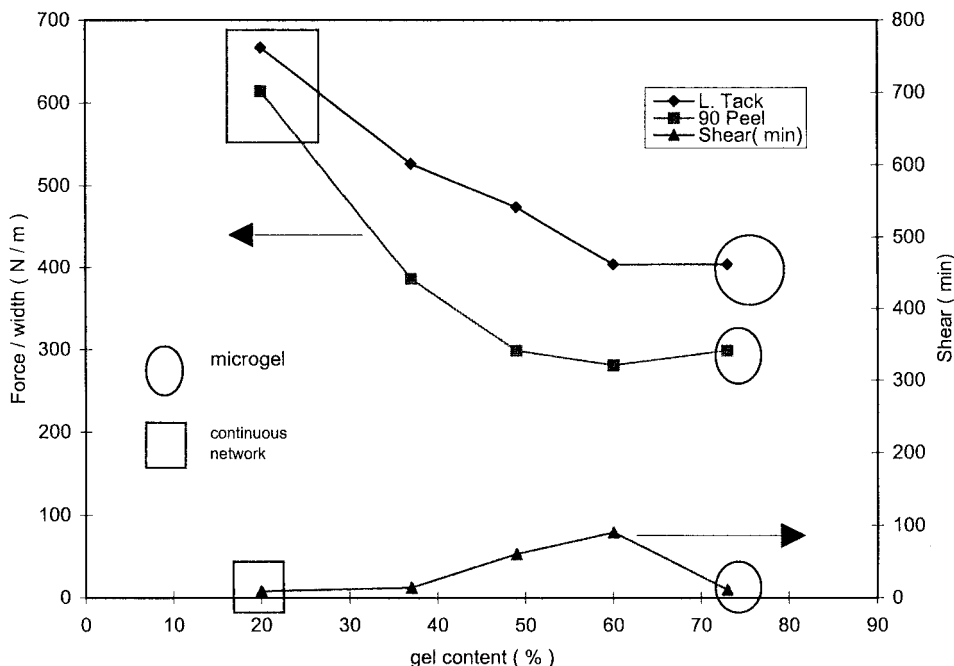


Figure 5 Adhesive performance of emulsion 1/emulsion 3 blends ( $M_c > M_e$ ).

tios as above. The films were cast and dried at the same conditions as above. Emulsion 5 had  $M_c > M_e$ , the same composition as that of emulsion 3, but 0.112 pphm APS was added late in the polymerization. Because APS was added late in

the polymerization, it is hypothesized that most of it would be located near the particle surface. As shown in Figure 6, there was an increased synergistic effect in shear strength compared to that found in emulsion 1/emulsion 3 blends. This sup-

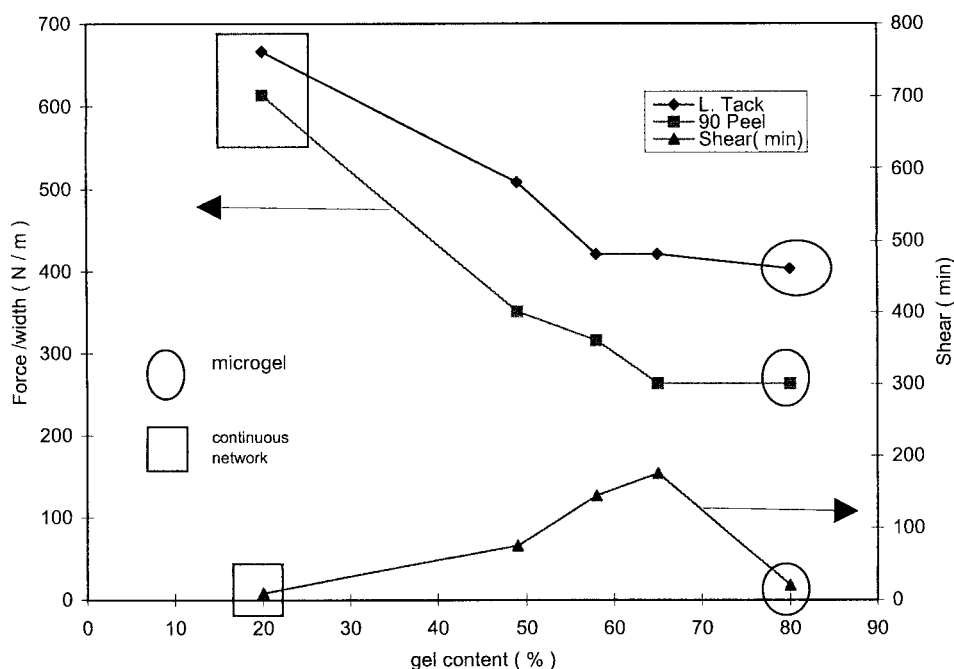
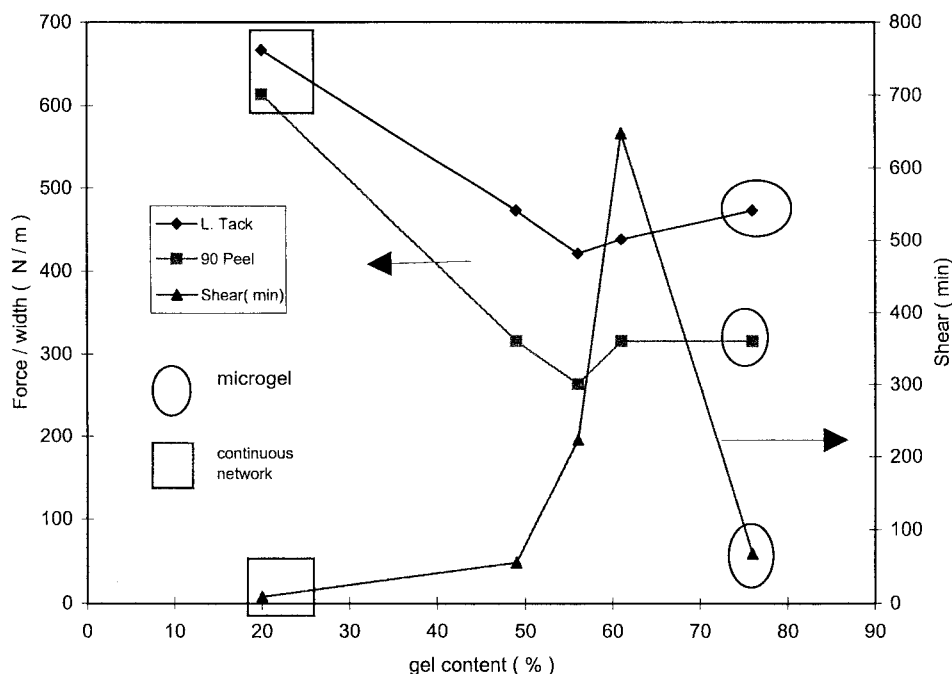


Figure 6 Adhesive performance of emulsion 1/emulsion 5 blends ( $M_c > M_e$  and APS added late in polymerization).



**Figure 7** Adhesive performance of emulsion 1/emulsion 6 blends ( $M_c > M_e$  and 4% AA).

ports the postulate that interlinking the microgels could significantly increase the shear.

Another way to increase the microgel reactivity involves increasing the level of AA in the copolymer. Figure 7 shows loop tack, peel, and shear off stainless steel of emulsion 1/emulsion 6 blend films plotted against the gel content. The films were cast and dried at the same conditions as above. A very strong synergistic effect in shear was observed. At 25/75 emulsion 1/emulsion 6 weight ratio, the PSA had shear 10 times higher than that of emulsion 6 and 72 times higher than that of emulsion 1. This result strongly confirms

the usefulness of linking the microgels with the linear polymer to increase shear.

#### Adhesive Performance of Emulsion Blends Versus Single Emulsion Versus Solvent Borne Acrylic PSAs

Table III shows adhesive performance of the emulsion blend versus single emulsion system versus solvent borne acrylic PSAs. The emulsion blend was 25/75 emulsion 1/emulsion 6. The single emulsion system was P(2EHA-*stat*-AA-*stat*-IBMA) 97/2.5/0.5 by weight synthesized by semi-continuous emulsion polymerization using V-50®

**Table III** Adhesive Performance of Emulsion Blend Versus Emulsion Versus Solvent Borne Acrylic PSAs

Adhesive	Gel Content (%)	L. Tack SS (N/m)	L. Tack HDPE (N/m)	90° Peel SS (N/m)	90° Peel HDPE (N/m)
Neat					
Em. 1/Em. 6 (25/75)	61	439	263	316	140
Emulsion <sup>a</sup>	70	351	210	210	123
Solvent borne <sup>b</sup>	69	368	228	193	88
15 wt % tackifier					
Em. 1/Em. 6 (25/75)	61	614	351	351	228
Emulsion <sup>a</sup>	70	456	246	281	158
Solvent borne <sup>b</sup>	61	807	263	491	193

<sup>a</sup> 2EHA/AA/IBMA 97/2.5/0.5.

<sup>b</sup> 2EHA/AA 97.5/2.5 with 0.75% A1 acetyl acetate.



initiator at 60°C.<sup>10</sup> The solvent borne acrylic was P(2EHA-*stat*-AA) 97.5/2.5 by weight crosslinked by postaddition of 0.75 wt % AAA.

In addition to the emulsion blend, single emulsion systems containing both gel and sol fraction within the same latex particle were synthesized. Therefore, the gel to sol ratio could not be varied independently without affecting the molecular weight of the linear polymer. In addition, in a single emulsion system, part of the linear polymer could be entrapped inside the microgels during polymerization because of the broad polydispersity effect early in the reaction. The higher molecular weight fraction would be more prone to attack by the propagating polymer radicals, resulting in branching and crosslinking with the lower molecular weight fraction entrapped inside the microgels due to the higher probability of hydrogen abstraction from the tertiary carbons. It is expected that a single emulsion system would have lower peel and loop tack compared to the emulsion blend due to lower sol fraction that is located outside the microgels.

In neat acrylic PSAs, the emulsion blend showed higher peel and loop tack compared to the single emulsion systems while retaining high shear, as shown in Table III. Furthermore, the emulsion blend showed higher peel, loop tack, and shear strength compared to solvent borne acrylic PSA.

In tackified acrylic PSAs, however, the emulsion blend showed a significant drop in shear holding power compared to the neat PSA, and the decrease was more pronounced than in the single emulsion system. This could be explained by the competitive processes of diffusion of the linear polymer into the tackifier molecules versus diffusion of the linear polymer into the microgels. The thermodynamic driving force was expected to favor the former process because the tackifier was composed of small molecules. Hence, the linear polymer-tackifier mixture, which now had higher  $T_g$ , would be too slow to diffuse into the microgels and would be self-crosslinked instead. Interlinking of the microgels was therefore much less probable than the formation of a physical network. In contrast, a single emulsion system already had the linear polymer entangled with the microgel to begin with, and therefore tackifier presented less difficulty in interlinking the microgels by the linear polymer.

## CONCLUSIONS

An emulsion blend of high-gel containing latex and gel-free latex could bring about a synergistic effect in increasing the shear holding power of the PSA film provided that  $M_c$  of the microgels was greater than  $M_e$  of the linear polymer, and  $M_w$  of the linear polymer was greater than  $2M_e$ . This synergism was a result of interlinking the microgels by the linear polymer due to the reaction of IBMA in the linear polymer and AA in the microgels when the film was heated. The reactivity of the microgels could be further increased by adding APS late in the polymerization or by increasing the AA level, resulting in much higher shear holding power of the emulsion blend film compared to that of the constituents.

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