

# Rheological and Adhesion Properties of Acrylic Pressure-Sensitive Adhesives

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**ABSTRACT:** Different pressure-sensitive adhesives (PSAs) based on acrylic monomers were synthesized under different reaction conditions. The synthesized PSAs have good adhesive properties and without leaving any residue can be easily peeled off from the surface of a substrate. The relationship between PSAs rheological behavior and its adhesion properties (e.g., peel, tack, and shear resistance) has been studied at constant adhesive thickness. The samples were examined for their surface energy and viscoelastic characteristics. It was observed that increase in reaction temperature and reaction time results in decreased storage modulus due to lowered molecular weight, which finally leads to lower elasticity of the PSA. While the storage ( $G'$ ) and loss ( $G''$ ) modulus of samples increase with increased initiator concentration, the elasticity of PSA is increased as

well. High  $G''$  at high frequency (100 Hz) represents high peel strength because of higher dissipation of viscoelastic energy during debonding. The tack values increase by lowering storage modulus at 1 Hz due to higher  $M_c$ . Shear values are increased by higher storage modulus at low frequency (0.1 Hz) due to hydrogen bonding of the different components. Some parallel investigations on the surface energy of the samples showed that they have different properties because of the nature of different monomeric units with their corresponding orientations. Our results reveal that the peel strength is not affected by surface energy.  
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**Key words:** pressure-sensitive adhesives; rheology; peel; tack; shear; adhesion

## INTRODUCTION

Pressure-sensitive adhesives (PSAs) are viscoelastic materials that can adhere to surfaces upon application of light contact pressure and leave no residue when they are removed.<sup>1</sup> Among different base polymers used in making PSAs, alkyl acrylates have enjoyed the fastest growth in commercial applications.<sup>1</sup> The acrylic PSAs are inherently tacky without any additional compounding. Their properties can be easily changed and modified by incorporating different monomers during polymerization and diffusion of enhancers.<sup>2</sup> The adhesion properties are characterized via measurements of three basic applicative properties; tack (the ability of the adhesive to adhere quickly under light pressure), peel adhesion (ability to resist its removal by peeling), and shear resistance or cohesion (ability to maintain its positioning under shearing forces).<sup>3</sup> These properties are not inherent properties of a PSA but a response to the adhesive's bulk and surface properties (in particular the surface energies of adhesive and adherend).<sup>4</sup> The viscoelastic nature of PSA controls its ad-

hesion to the substrate surface and determines the duration of its application. It also enables PSA to exhibit both solid- and liquid-like behavior depending on variables such as temperature and frequency of the applied stress. This behavior of PSA is described by viscoelastic parameters such as storage (elastic) modulus ( $G'$ ), and loss (viscous) modulus ( $G''$ ). The elastic modulus describes the solid-like character, whereas, the loss modulus describes the liquid-like character of the adhesive. Therefore, a correlation exists between the degree of adhesion and the viscoelastic properties of PSAs.<sup>4</sup> Tack or bonding is a low rate process where the adhesive should be liquid-like and hence able to flow sufficiently to promote intimate contact between itself and the substrate. Peel or debonding is a high rate process where the adhesive should be solid-like, i.e., cohesive and internally strong. Also the shear resistance or holding power is a low rate process at high deformation and it is influenced by the storage (elastic) and loss (viscous) moduli and other factors, e.g., the friction coefficient between the adhesive and the adherend i.e., cohesive strength.<sup>4,5</sup> The interpretation of these characteristics in terms of oscillatory viscoelastic parameters demonstrate that low oscillatory frequencies are associated with tack, whereas, high frequencies are associated with the peel process. Therefore, the loss modulus should predominate ( $G'' > G'$ ) at low frequencies, and the storage modulus should

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**TABLE I**  
Effect of Different Reaction Temperatures (Time: 3 h, AIBN: 1%)

Code	$T$ (°C)	$T_g$ by DSC (°C)	$M_w \times 10^5$ (g/mol)	Peel (N/25 mm)	Tack (N/mm <sup>2</sup> )	Shear resistance (min)
PSA1	70	-41	5.60	14.5 ± 0.1	4.7 ± 0.3	2.5 ± 0.4
PSA2	80	-50	4.36	8.6 ± 0.2	4.6 ± 0.1	0.5 ± 0.08
PSA3	90	-47	1.82	2.4 ± 0.05	3.6 ± 0.05	0.4 ± 0.08

predominate ( $G' > G''$ ) at high frequencies.<sup>4</sup> According to literature, it was described how the moduli at high frequency are related to peel or quick stick tests, and at low frequencies, to shear resistance of adhesives.<sup>6</sup> On the other hand, the chemical structure of a PSAs base polymer plays an important part in modulus determination. Linear chain backbones, without side groups to restrict rotation, are very flexible and tend to exhibit low modulus.<sup>7</sup> Several authors have recently reported that the rheological behavior of copolymer-based adhesives show strong links with adherence properties. The first phenomenological postulate on PSA characteristics was provided by Dahlquist.<sup>8</sup> PSA materials were those that had creep compliance  $>10^{-6}$  cm<sup>2</sup>/dyn. According to Dahlquist's postulate, the need of rapid viscous flow to achieve intimate contact between the substrate and PSA, providing that the PSA is able to wet the substrate, does not consider the viscoelastic energy dissipation during peeling of a PSA tape.<sup>2</sup> In the case of hot-melt PSAs based on block copolymers, it was demonstrated that the ratio between the triblock and diblock copolymers is a key parameter to control their rheological behavior.<sup>7</sup>

Amaral<sup>9</sup> showed that both the rheological and adhesive properties were found to be very dependent on the initial latex particle size distribution in acrylic PSA prepared from high solid emulsions and demonstrated that latices with different particle size distributions gave significantly higher adhesion energies and clear evidence of a fibrillar detachment process. In studies by Jensen et al.<sup>10</sup> on PSAs prepared from polypropylene oxide, relationship between the viscoelastic parameters and the PSA peel properties is found that the resistance to peel is proportional to the peel strip dimensions and to the dissipated energy during deformation and thus the loss modulus at the peel frequency. In studies of the linear viscoelastic behavior of PSAs and the energy involved during

peeling, it is found that where the surface energy is small, the peel strength is proportional to the ratio of  $G''/G'$ , where  $G''$  is measured at the peel frequency and  $G'$  at the bonding frequency.<sup>10</sup>

In this study, a detailed investigation on rheological parameters was performed because the rheological behavior of acrylic copolymers is the main factor in development of PSA applications. As part of a more comprehensive study, in a previous work, acrylic PSAs were synthesized using acrylic monomers such as 2-ethylhexyl acrylate (2-EHA), hydroxyethyl acrylate (HEA), glycidyl methacrylate (GMA), and vinyl acetate (VAc), under different reaction conditions, and some correlations were found between the peel strength and tack properties with molecular weight.<sup>1</sup> According to the best of our knowledge, there is no published report regarding the relationship between the rheological properties, surface energy, and adhesion properties of these polymers. The complex correlations are fully explored for the synthesized acrylic PSAs. It is proved that elasticity and viscoelastic properties of synthesized acrylic PSAs are affected under different polymerization conditions. On the other hand, it is observed that, in all synthesized PSAs, there is a weak correlation between the peel strength and surface energy for long contact times.

## EXPERIMENTAL

### Materials

2-Ethyl hexyl acrylate (2-EHA, 99%), vinyl acetate (VAc, >99%), and ethyl acetate were purchased from Merck (Germany, Darmstadt); 2-hydroxy ethyl acrylate (2-HEA, >97%), glycidyl methacrylate (GMA, >97%) and 2,2' azobisisobutyronitrile (AIBN) as initiator were purchased from Fluka (Switzerland, Buchs). Polyester/ethyl vinyl acetate (Scotch Pak9732) as a backing layer with 85 μm thickness (3

**TABLE II**  
Effect of Different Reaction Times ( $T$ : 70°C, AIBN: 1%)

Code	$t$ (h)	$T_g$ by DSC (°C)	$M_w \times 10^5$ (g/mol)	Peel (N/25 mm)	Tack (N/mm <sup>2</sup> )	Shear resistance (min)
PSA1	3	-41	5.60	14.5 ± 0.1	4.7 ± 0.3	2.5 ± 0.4
PSA4	6	-49	4.44	16.3 ± 0.2	4.5 ± 0.1	0.8 ± 0.1
PSA5	9	-52	5.33	17.5 ± 0.3	4.3 ± 0.1	>100 ± 0.1

**TABLE III**  
Effect of Different Initiator Concentrations (Time: 3 h, T: 70°C)

Code	AIBN (%)	$T_g$ by DSC (°C)	$M_w \times 10^5$ (g/mol)	Peel (N/25 mm)	Tack (N/mm <sup>2</sup> )	Shear resistance (min)
PSA6	0.25	-43	7.65	13.6±0.7	4.2±0.08	1.6±0.3
PSA7	0.5	-43	6.70	15.6±0.5	4.4±0.08	4.5±0.4
PSA1	1	-41	5.60	14.5±0.1	4.7±0.3	2.5±0.4

M, MN) was used. All chemicals of reagent grades were used without further purification.

### Preparation of acrylic PSAs

Acrylic PSAs were prepared by using 67% 2-ethyl hexyl acrylate (2-EHA), 28% vinyl acetate (VAc), 4.9% hydroxy ethyl acrylate (HEA), and 0.1% glycidyl methacrylate(GMA) by free radical polymerization in ethyl acetate. The polymerization reaction was carried out under various reaction conditions like different reaction temperatures, initiator concentrations, and reaction times.<sup>1</sup> The varying reaction conditions such as temperature (PSA1, PSA2, and PSA3), reaction time (PSA1, PSA4, and PSA5), and initiator concentration (PSA6, PSA7, and PSA1) are shown in Tables I–III. Then an appropriate amount of resulting acrylic product as PSAs was coated onto a backing layer by film applicator (elcometer 3580) at a controlled thickness. For each formulation of the synthesized adhesive three samples were prepared. The coated products at first were kept at room temperature for 20 min and then placed in an oven at 50°C for 45 min to drive off all volatile processing solvent. Then adhesion properties were investigated for all coated samples by peel strength, tack, and shear tests.

### Instrumental analysis

#### Gel permeation chromatography

The average molecular weight of the optimized copolymer was determined using gel permeation chromatography (Agilent 1100 series, Santa Clara, USA) at ambient temperature. A sample in tetrahydrofuran was placed in Agilent 1100 series, consisting of a refractive index detector RI. The column used was Agilent PL gel 10  $\mu$ m.

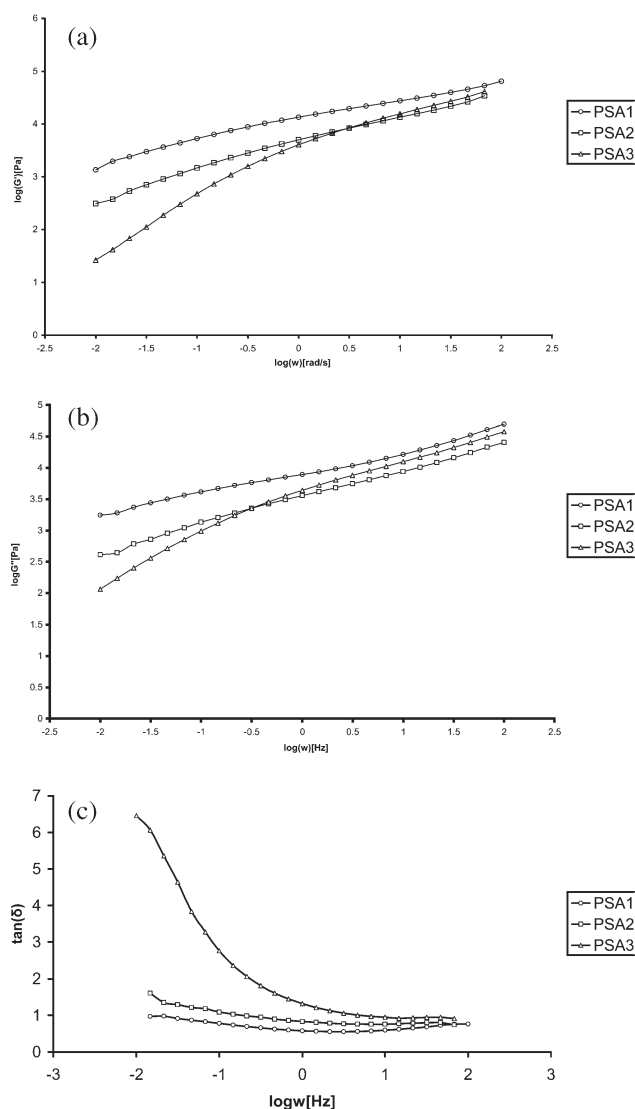
#### 180° peel test

Peel tests were carried out according to ASTM D3330 on adhesive-coated tapes with 25 mm width. Dried PSA tapes with 45  $\mu$ m thickness were pressed between stainless steel plates by a 5-kg rubber roller passing twice over the samples. After preparation of PSA tape/stainless steel joints, they were stored at room temperature for 20 min. Peel force in 180°C

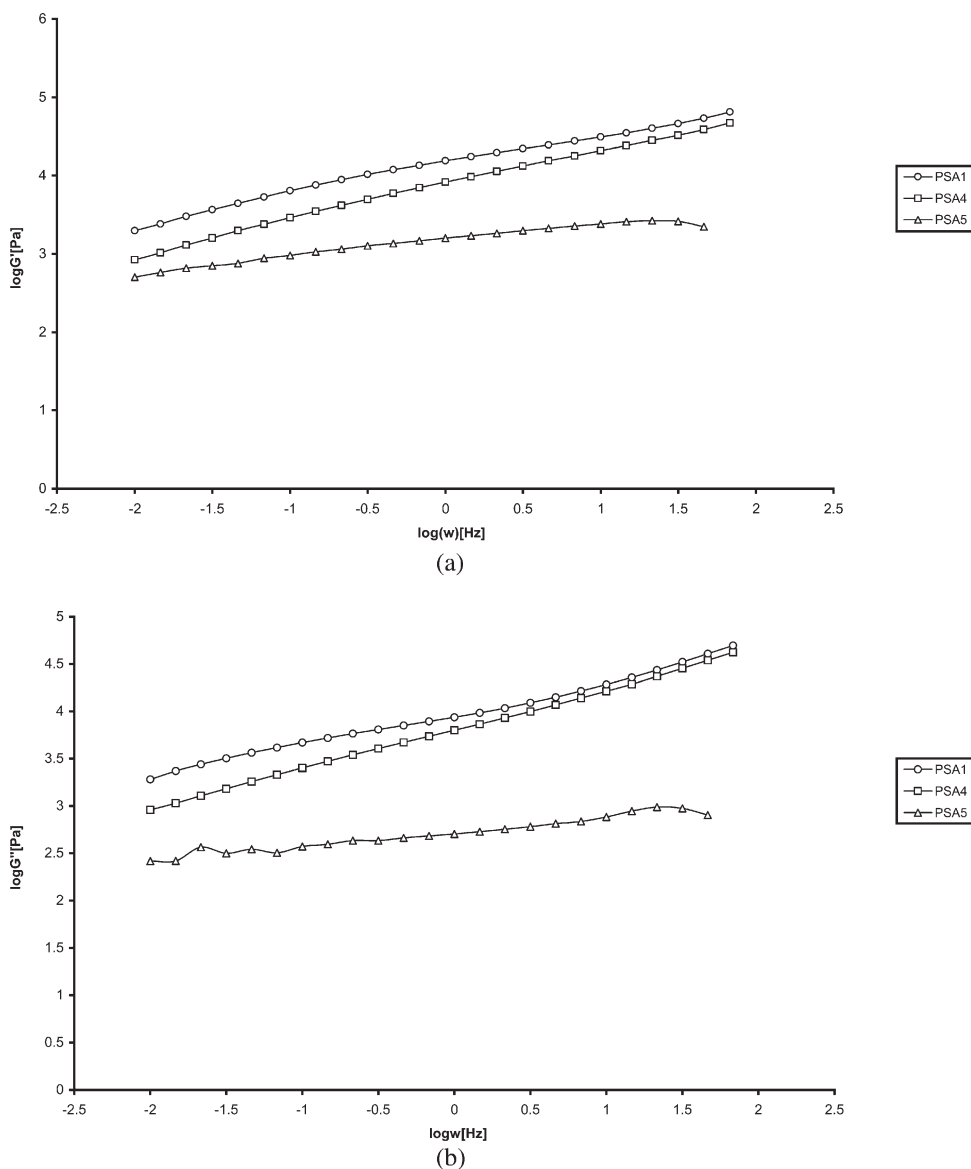
direction was measured for at least three samples at a peel rate of 30.5 cm/min at room temperature using a ChemInstruments adhesive/release tester AR-1000 (Fair Field, OH, USA).<sup>11</sup>

#### Probe tack

Tack tests were carried out on adhesive tapes each with 45  $\mu$ m thickness. According to ASTM D3121,



**Figure 1** The plots of: (a) storage modulus ( $G'$ ), (b) loss modulus ( $G''$ ), and (c) damping factor of synthesized adhesives at different reaction temperatures.



**Figure 2** The plots of: (a) storage modulus ( $G'$ ), (b) loss modulus ( $G''$ ) of synthesized adhesives at different reaction times.

the ChemInstruments probe-tack PT-500 (fair field, OH) tester was applied on at least four samples.<sup>11</sup>

#### Shear resistance test

Shear resistance tests were carried out according to the ASTM D3654 on stainless steel with strips (15 mm  $\times$  60 mm) of the above adhesive-coated films. The steel substrates were washed with acetone and dried at 50 °C to remove impurities. One end of each adhesive strip was joined to the stainless steel with a contact area of 15 mm  $\times$  15 mm, whereas the other end was connected to a 0.5 kg hanging weight.<sup>12</sup> Shear resistance was measured on at least three samples at room temperature using ChemInstruments shear tester HT-08 (Fair Field, OH).

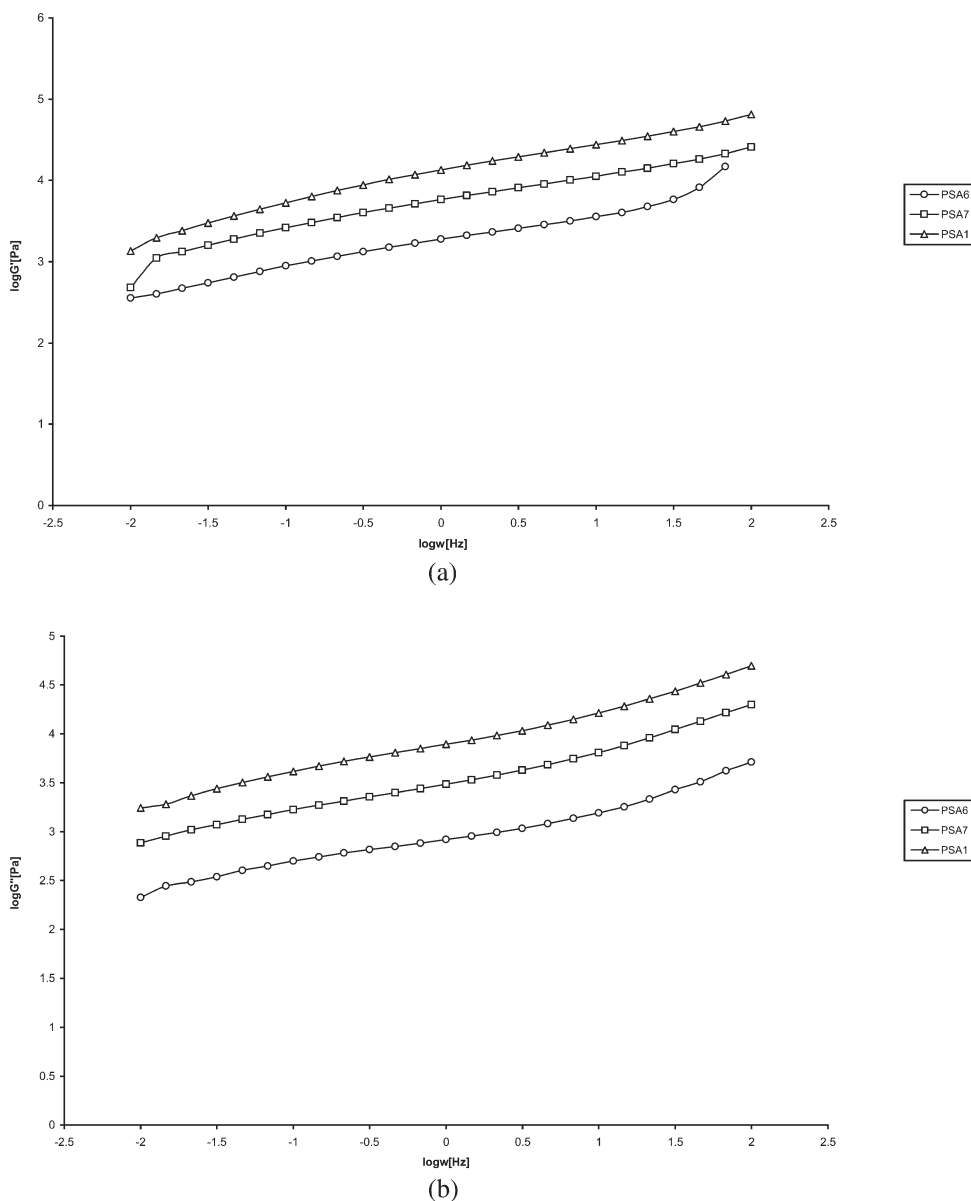
Specifications of the synthesized PSAs at various reaction parameters are summarized in Tables I–III.

#### Contact angle measurement

For estimation of the surface energy, contact angles were determined at room temperature using distilled water and diiodomethane by a contact angle measuring system Gio (Kruss, Germany). Dispersion and polar components of the surface energy,  $\gamma_A^d$  and  $\gamma_A^p$  were determined according to the improved Owens method.<sup>13</sup>

#### Rheological studies

Viscoelastic properties were determined on a rheometer MCR-300 (Anton Paar-Physica, Austria, Graz).



**Figure 3** The plots of: (a) storage modulus ( $G'$ ), (b) loss modulus ( $G''$ ) of synthesized adhesives at different initiator concentrations.

The measurements were carried out by parallel plate method. Oscillation frequency was varied from 0.01 to 100 rad/s at different strains and temperature of 25°C at which each frequency sweep was run separately.  $G'$  and  $G''$  are plotted versus frequency at room temperature.<sup>13</sup>

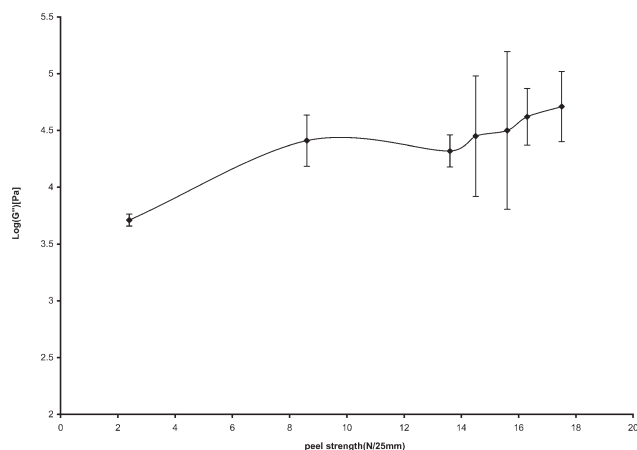
## RESULTS AND DISCUSSION

### Dynamic viscoelastic properties

All PSAs are viscoelastic liquids (semisolids). The moduli can be divided into in-phase ( $G'$ ) and out-of-phase ( $G''$ ) components in conventional manner and plotted as a function of frequency  $\omega$  (from 0.01 to 100 Hz). The dynamic mechanical properties (as a

function of frequency,  $\omega$ ) have already been correlated to adhesive properties.<sup>13</sup>

The plots of storage modulus ( $G'$ ) and loss modulus ( $G''$ ) at different reaction parameters as functions of angular frequency (0.01–100 Hz) are shown in Figures 1–3. It was found that at higher reaction temperature and time, the storage and loss modulus decrease, indicating that the elasticity of PSAs is decreased due to decreasing molecular weight and increased entanglements. While with increased initiator content the storage and loss moduli increased, indicating that PSAs higher elasticity is possibility due to chain transfer and restricted polymer chain mobility.<sup>2,5</sup> Also the sample has higher  $G'$  and a lower  $\tan \delta$  which shows that the PSA has a higher cohesive strength.<sup>2,5</sup>



**Figure 4** Plot of the logarithm of loss modulus ( $G''$ ) in frequency 100 Hz against peel strength.

Viscoelastic properties of an adhesive play an important role in adhesion properties. In a good PSA system, the viscoelastic properties must balance each other. This balance would include sufficient flow enabling the adhesive to form a bond with substrate.<sup>14</sup> According to Tobing and Klien<sup>2</sup> studies, because both tack and peel are the outcome of viscoelastic processes, they bear direct relationship with the molecular weight ( $M_w$ ) of the polymer and its entanglements. Very low  $M_w$  compositions, although produce high viscous flow because of plasticization effect, do not necessarily result in high viscoelastic energy dissipation (VED) during debonding when filaments might fracture very quickly in the absence of entanglements. Hence, high viscoelastic energy dissipation can only be obtained if there is a sufficient anchorage of the adhesive onto the substrate

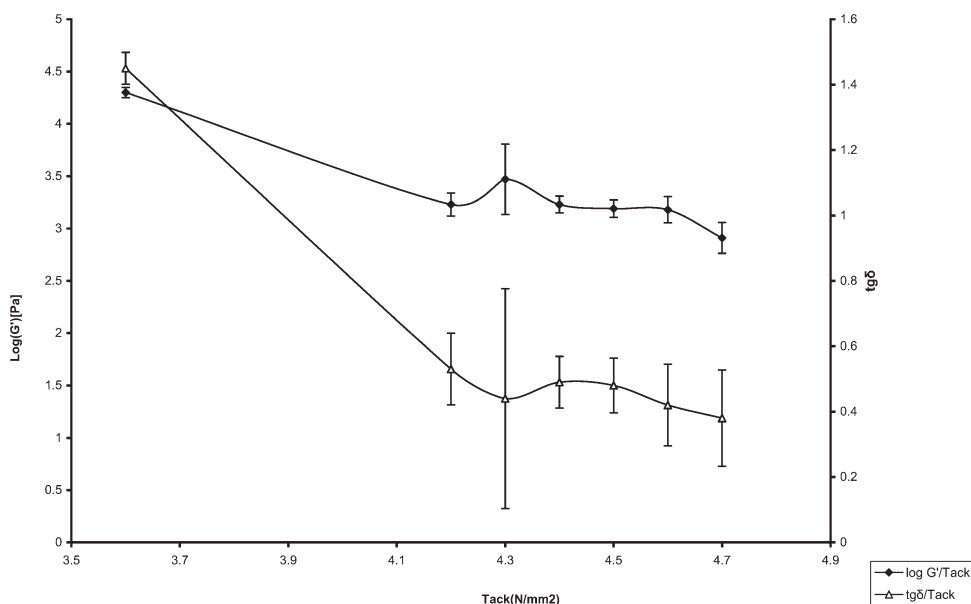
and low modulus of high elongation-at-break fibrils that are deformed during the peeling process. When the PSA tape is peeled off from a substrate, large viscoelastic energy dissipation must occur to give high-peel energy. Lower bound approximation in relating peel energy with viscoelastic energy dissipation and work of adhesion ( $W_A$ ) can be expressed as follows<sup>11</sup>:

$$\text{Peel energy} \propto W_A[1 + \text{V.E.D}] \quad (1)$$

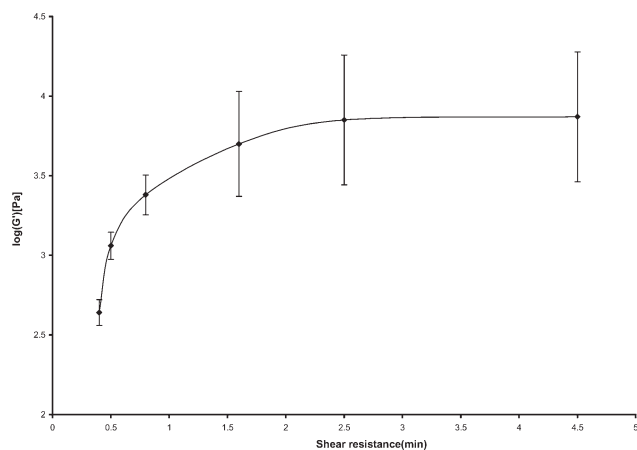
Peel energy is related to the viscous dissipation phenomena characterized by the loss modulus ( $G''$ ). In this study, the effect of loss modulus on the peel behavior of the synthesized adhesives was investigated. The loss modulus ( $G''$ ) versus peel strength at frequency 100 Hz are shown in Figure 4 for all samples. It was observed that the peel strength of samples increase with increased loss modulus, indicating that the samples with higher  $G''$  would dissipate higher viscoelastic energy during debonding process. On the other hand, the storage modulus ( $G'$ ) of PSA was related to the diffusion property of the bulk adhesion onto the substrates within a short period of time.<sup>5</sup> Zosel<sup>15</sup> investigated that the debonding energy (related to  $G''$ ), in a peel or tack test, is proportional to the average mass between polymer chain entanglements ( $M_e$ ). Also  $M_e$  maybe related to  $G'$  with respect to the following equation<sup>16</sup>:

$$M_e = \rho RT / G_n^0 \quad (2)$$

where  $\rho$  is the density of polymer or blend;  $R$  is 8.31 J/mol K,  $T$  is absolute temperature, and rubbery plateau modulus  $G_n^0$  is determined from  $G'$  at the onset



**Figure 5** Plot of the logarithm of storage modulus ( $G'$ ) at frequency 1 Hz against tack values.



**Figure 6** Plot of the logarithm of storage modulus ( $G'$ ) at frequency 0.1 Hz against shear resistance.

of rubbery region. In Figure 5, the storage modulus ( $G'$ ) and  $\tan \delta$  ( $\tan \delta = G''/G'$ ) are shown at frequency 1 Hz versus tack values of the samples. It is observed that the tack values increase with decreased storage modulus. Therefore, according to Eq. (2), lowered  $G'$  corresponds to higher value of  $M_e$ . This means that the adhesive cannot be deformed easily and develop good efficient contact with the substrate which leads to increased tack value. On the other hand, it was observed that higher tack value is obtained at lower storage modulus, a characteristic of a good PSA system that makes bonding favorable even on rough surfaces.<sup>9</sup>

Shear resistance, unlike peel and tack, is directly proportional to zero-shear viscosity at room temperature, which in turn is influenced by entanglement, crosslinking, and also morphology. The higher entanglement and crosslinking densities, the higher would be the zero shear viscosity, and hence the higher shear resistance.<sup>2</sup> Therefore, the shear resistance at low frequency is studied and Figure 6 shows the storage modulus ( $G'$ ) versus shear resistance at frequency 0.1 Hz for the samples. It is observed that shear resistance increases with increased storage modulus.

### The effect of surface energy on adhesion properties

Although proper viscoelasticity is a prerequisite for practical adhesion of PSAs, it is not the only factor that affects adhesion. For adhesion to many substrates, the surface energy of the adhesive layer becomes important. To be effective, a PSA must immediately wet a surface as soon as they are brought into contact. Therefore, the relationship between the surface energy of the adherend and surface energy of the adhesive becomes critical.<sup>13,17</sup> Hereby, the effect of surface energy on adhesion properties is evaluated and the results are summarized in Table IV. However, the initial amount of monomers is constant in all formulations and due to the differences in monomer reactivity ratios, their conversion is changed and therefore the final copolymer compositions (the nature of monomeric units and their orientations, etc.) are different.<sup>1</sup> Considering the presence of hydrophilic and hydrophobic monomers in the final copolymer composition, the surface energies are predictably changed.

Separation of an adhesive tape from a substrate is a process in which thermodynamic work of adhesion and dissipation factors are both involved. In other words, the total work of peeling may be expressed in the following form<sup>18</sup>:

$$W_T = W_A(1 + \phi) \quad (3)$$

where  $W_A$  and  $W_T$  are the respective thermodynamic and total works of adhesion, and  $\phi$  is a dissipation factor. Thermodynamic or reversible work of adhesion,  $W_A$ , is the change in free energy when the materials are brought into contact, and it is the same as the amount of work expended under reversible or equilibrium conditions to disrupt the interface.  $W_A$  is related to surface-free energies or surface tensions by the Dupre equation.<sup>18</sup> When phases separate out in dry air, the following equations may hold:

$$W_A = \gamma_A + \gamma_S - \gamma_{AS} \quad (\text{Dupre' equation}) \quad (4)$$

**TABLE IV**  
Contact Angles and Surface Energy of Pressure-Sensitive Adhesive Tapes

Sample	Contact angle (°)		$\gamma$ (mN/m)		$W$ (mN/m)			Peel (N/25mm)
	Water	Diiodo methane	$\gamma^d$ (mN/m)	$\gamma^p$ (mN/m)	$W_A$	$W_T \times 10^3$	$W_T/W_A$	
PSA1	122.8	86.2	14.45	0.02	39.81	1160	29136.3	14.5 ± 0.1
PSA2	121.7	73.2	21.10	0.35	51.89	704	13566.8	8.6 ± 0.2
PSA3	112.6	62.2	27.32	0.08	55.54	192	3457.1	2.4 ± 0.05
PSA4	120.7	81.6	16.69	0.03	42.97	1304	30340.8	16.3 ± 0.2
PSA5	124.5	89.4	12.95	0.02	37.76	1400	37078.6	17.5 ± 0.3
PSA6	121.3	86.2	14.44	0.00	38.53	1088	28238.9	13.6 ± 0.7
PSA7	120	82.3	16.35	0.01	41.90	1224	29215.1	15.6 ± 0.5
Steel	56.3	48.7	25.7	20.2	—	—	—	—

As

$$\gamma_{AS} = \gamma_A + \gamma_S - 2[(\gamma_A^d \gamma_S^d)^{1/2} + (\gamma_A^p \gamma_S^p)^{1/2}] \quad (\text{Fowkes eq}) \quad (5)$$

Then

$$W_A = 2[(\gamma_A^d \gamma_S^d)^{1/2} + (\gamma_A^p \gamma_S^p)^{1/2}] \quad (6)$$

where  $\gamma_A^d$ ,  $\gamma_S^d$ ,  $\gamma_A^p$ , and  $\gamma_S^p$  are disperse and polar parts of adhesive and substrate. These parameters, calculated by contact angles in addition with  $W_A$ ,  $W_T$ , and  $\phi$ , are shown in Table IV.  $W_T$  has been calculated using the following equation:

$$W_T = F/b(1 - \cos \Theta) \quad (7)$$

where  $F$ ,  $b$ , and  $\Theta$  are peel energy, tape width, and peel angle, respectively, and  $W_A$  is very small compared to  $W_T$  and  $\phi$  (viscoelastic dissipation factor).

According to Table IV, it is found that in all synthesized PSA, there is a weak dependence between peel and surface energy for long contact times (about 1200s).  $W_A$ , resulting from the dissociation of the Van der Waals bonds at the interface, can be several orders of magnitude smaller than viscoelastic energy dissipation. Then the effect of  $W_A$  on peel strength can be neglected. The same result was obtained by Zosel.<sup>11</sup>

## CONCLUSIONS

Since adhesion properties of PSAs are responsive properties of the bulk and surface of the adhesive, thus investigation of viscoelastic behavior is considered highly important. As a result, we have focused on the relationship between rheological and adhesion properties. The modulus data at frequency range of 0.1–100 Hz describe the bonding and

debonding behavior of PSAs. It was observed that, for synthesized acrylic PSAs, the changes in peel strength at higher frequency (100 Hz) tack values at frequency of 1 Hz and shear resistance at low frequency (0.1 Hz) obey general rules. To this end, we have clearly shown that there is a strong dependence between peel strength and viscoelastic energy dissipation and the effect of work of adhesion on peel strength can be neglected.

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