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Thermochimica Acta 391 (2002) 245–255

thermochimica acta

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The characterization of polyvinyl butyral by thermal analysis

A.K. Dhaliwal, J.N. Hay*

The School of Metallurgy and Materials, The University of Birmingham, Edgbaston, P.O. Box 363, Birmingham B15 2TT, UK

Received 4 September 2001; received in revised form 15 January 2002; accepted 30 January 2002

Abstract

A range of thermal analytical techniques have been used to characterize PVB obtained from various manufacturers and recycled material recovered from glass recycling units. ^{13}C NMR spectroscopy was used to confirm that PVB is a random ter-polymer containing butyral and hydroxyl side groups with a small amount of acetate units. Little variation in chemical composition was observed between the grades studied. The PVB also contained between 20 and 25 wt.% of plasticizers and GC mass spectroscopy has been used to determine that the dominant plasticizer was dibutyl sebacate. A more convenient method to determining the amount present, however, was differential scanning calorimetry since PVB being an amorphous rubber its glass transition temperature, T_g, varied with plasticizer content. Most recycled PVB samples had similar T_g's but these could be adjusted by addition of further amounts of dibutyl sebacate.

PVB decomposed by side group elimination above 260 °C. Butyraldehyde, butenal, water, and acetic acid were the major components from these elimination reactions, but benzene and other aromatic products were observed from the decomposition of polyene residues. Below 260 °C the change in mass was attributed to plasticizer loss by the evaporation of dibutyl sebacate from the PVB matrix. It was concluded that recycled and blended grades of PVB could be re-used as a laminating agent for glass and that PVB should be recycled rather than placed in landfill.

Keywords: Polyvinyl butyral; Recycling; DSC; TG mass spectroscopy

1. Introduction

With increasing raw material costs and demands for pollution control becoming more important there is an incentive to recycle plastic waste [1,2]. Polyvinyl butyral (PVB) is extensively used in glass lamination, but it is not recycled in the UK because it is felt that the process is too demanding in requiring uniform control of polymer properties. Instead PVB from automotive sources is disposed off in landfill [3]. This is a waste of an expensive, valuable and non-renewable commodity as well as energy and land.

The main use of PVB is in safety glass laminates, particularly in automotive, aerospace and architectural glass. Worldwide 65% of all PVB is used in automotive applications. While the glass is recycled PVB is not. It is a disposable by-product of the glass recycling industry and available in quantity with no additional collection charges. An alternative to disposal in landfill is to recycle by mechanical means but central to this is an appreciation of the variation in molecular structure of PVB and its effect on material properties and end use.

PVB is a polyacetal produced by the condensation of polyvinyl alcohol with *n*-butyraldehyde in the presence of an acid catalyst [4]. The condensation

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* Corresponding author. Tel.: +44-121-414-4544; fax: +44-121-414-5232/4532.
E-mail address: j.n.hay@bham.ac.uk (J.N. Hay).

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PII: S0040-6031(02)00187-9

reaction produces 1,3-dioxane rings but it is not taken to completion leaving some unreacted hydroxyl groups which promote good adhesion to the glass substrate on lamination. Since polyvinyl alcohol is produced from the hydrolysis of polyvinyl acetate there is a limited amount of acetate groups also present. The final structure can be considered to be a random ter-polymer of vinyl butyral, vinyl alcohol and vinyl acetate, typically 76, 22 and 2%, respectively [5]. Variations in chemical composition can occur between manufacturers and the recycled products could vary in composition and properties depending on the original source.

PVB is also highly plasticized and different plasticizers could be present to different extents. In recycling PVB from different manufacturers the different plasticizers present may interact in a deleterious way and alter the properties of the PVB. Finally, weathering of the recycled PVB could hydrolyze the butyral groups, remove plasticizer by leaching and alter the nature of the functional groups of the polymer. All of these will change the final properties of the recycled PVB.

This paper deals with the characterization of PVB obtained from the glass recycling industry and the effect of structural variation observed in the material as measured by thermal analytical techniques on the material and mechanical properties of the product.

2. Experimental

Unused commercial PVB film, suitable for glass lamination was obtained from the manufacturer, see Table 1. Recycled material was recovered from the delamination of car windshields from known commercial products. Finally, blends of PVB were prepared using material from known sources; both recycled and commercial materials were used. Blending was carried out on a Schwabenthan two roll mill for 5 min at 140 and 150 °C and the crepe produced molded into $100 \times 100\ \mathrm{mm}^2$ rectangular sheets at 150 °C in a hydraulic press, Moore E1127. PVB was also obtained as mixed waste scrap from automotive glass from a glass recycling unit. Glass shards were removed by dissolving the polymer in ethanol (5 wt.%) and filtering through a column filled with fine sand. Films were produced by spin coating 10% solutions on to $16 \times 16\ \mathrm{mm}^2$ polypropylene plaques at 200 rpm for 30 min by solvent evaporation. The films readily lifted from the polypropylene plaques and were used after vacuum drying. Unused plasticized film was obtained from Sekisui, Dupont and Monsanto, as commercial samples used to laminate automotive glass. Samples used for each analysis were cut directly from the films. Un-plasticized material was obtained by Soxhlet extraction with *n*-hexane—a non-solvent for PVB.

Table 1
Source of PVB film

Polymer serial number		Manufacturer	Grade
Commercial	Recycled		
S-1	SR-1	Sekisui	S-Lec
D-1	DR-1	Dupont	Butacite
M-1	MR-1	Monsanto	Saflex
R-1		Richardson	Mixed waste

50:50, 33:33:33 blends of S-1, D-1 and M-1.

A Stanton–Redcroft Thermogravimetric Unit, STA-780, interfaced to a PC and a CPC-706 temperature programmer, was used to determine the mass loss from PVB samples from ambient to 800 K in flowing nitrogen, $40\ \mathrm{cm}^3\ \mathrm{min}^{-1}$. It was used dynamically at $10\ \mathrm{K\ min}^{-1}$ and isothermally after heating at $20\ \mathrm{K\ min}^{-1}$ to the required temperature. Ten to forty milligrams samples were used in a platinum crucible. Simultaneous mass spectroscopic analysis, TG-MS, was carried out on the volatile products using a Thermolab mass spectrometer from Fison Instruments.

Gas chromatography-mass spectroscopic (GC-MS) analysis was carried out on the material extracted from the polymer by Soxhlet extraction using a Carlo Erba GC 8000 series gas chromatrographic unit and a VG Prospec mass spectrometer. The chromatographic column was programmed heated at $10\ \mathrm{K\ min}^{-1}$ from 350 to 650 K.

A Perkin-Elmer differential scanning calorimeter, DSC-2, interfaced to a PC was used to measure the glass transition temperature of PVB, using experimental procedures outlined previously [6]. Sub-ambient temperatures were achieved by cooling the calorimeter block to 190 K using liquid nitrogen. The glass transition was determined at 10, 20 and $40\ \mathrm{K\ min}^{-1}$ heating rates and the value determined by extrapolating to zero rate.

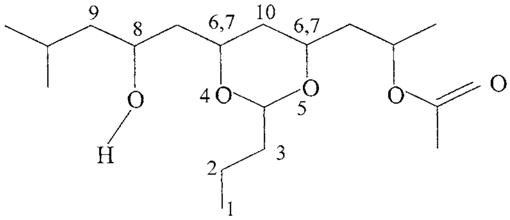
An attenuated total reflection attachment was used to measure the IR spectra of the polymer films using a

Nicolet 760 IR-Magna FT-IR spectrometer over the range 4000–700 cm^{-1}.

^{13}C NMR spectra were measured with a Jeol GX270 NMR Spectrometer operating at 67 MHz. The spectra were averaged over 250,000 scans at 295 K in dimethyl formamide solutions, containing deuterated dimethyl formamide as a lock.

3. Results and discussion

3.1. *The characterization of commercial PVB and plasticizers*



The ^{13}C NMR spectrum of PVB was complex containing 10 absorption bands characteristic of the various structural units present. These are labeled and assigned to the structural units in the chemical formula shown above following the format in the literature [7–9].

The chemical shifts and absorbance bands of each structural unit are listed in Table 2a. The major structural unit in all the samples studied and the mixed recycled waste was *n*-butyral groups (76 mol% *n*-butyral) with 22 mol% hydroxyl and the residue acetate units, see Table 2b. An exception was sample M-1, which had a significantly lower concentration of hydroxyl groups. The recycled waste did not have a significantly different ratio of functional units that could be attributable to reaction with the glass substrate during lamination or to hydrolysis on weathering.

Plasticizer was extracted from the recycled PVB samples with *n*-hexane. The extracts from the polymers were evaporated to low volume and analyzed by GC-MS. The polymer residue was vacuum dried to constant weight. Approximately 25% of the sample mass was removed by this procedure, see Table 3, but the recycled mix contained substantially less, typically 15%, indicating some loss during recycling. GC analysis of the extract showed that it contained four components, each with very different retention times and detector response. The first was due to trace

Table 2
(a) The chemical shifts and absorbance bands of each structural unit; (b) % molar composition of hydroxyl to butyral groups

	Structural unit	Chemical shift (ppm)				Relative intensity
		Polymer serial number S-1	Polymer serial number D-1	Polymer serial number M-1	Polymer serial number R-1	
(a) NMR assignments						
1	Butyral-CH_3	18.19	18.12	18.12	18.17	23.0
2	Butyral-CH_2	21.99	21.98	21.99	21.94	28.5
3	Butyral-CH_2	41.63	41.62	41.63	41.61	28.5
4	*meso*-Butyral-CH	105.5	105.6	105.6	105.5	100.0
5	Racemic-butyral-CH	98.56	98.58	98.59	98.65	11.5
6	*meso*-Butyral-CH	78.87	78.85	78.88	78.86	52.6
		77.54	77.49	77.51	77.53	
		76.58	76.48	76.43	76.46	
7	Racemic-butyral-CH	72.49	72.51	72.49	72.50	13.9
8	Vinyl-OH–CH	69.06	68.76	69.01	68.70	20.5
9	Vinyl-OH–CH_2	48.90	48.87	48.92	49.03	12.5
		49.42	49.40	49.44	49.37	
10	Butyral-CH_2	41.15	41.46	41.14	41.69	32.9
		46.84	46.82	46.85	46.68	
(b) % Molar composition of hydroxyl to butyral groups						
	Hydroxyl	22.4	22.1	7.9	23.5	
	n-Butyral	77.6	77.9	92.1	76.5	

Table 3
Hexane extracted material from PVB samples

Polymer serial number	% Mass extracted
M-1	24 ± 3
S-1	25 ± 3
D-1	21 ± 3
R-1	15 ± 3

Table 4
Glass transition temperature of PVB samples

Polymer serial number	Glass transition temperature (K)
S-1	290 ± 2
D-1	289 ± 2
SR-1	289 ± 2
DR-1	289 ± 2
R-1	285 ± 2
Un-plasticized PVB	347 ± 2

amounts of hexane while the second and fourth were present in greatest amounts. The third was present only in trace amounts. From the top mass peak and fragmentation patterns using both chemical impact and electron impact mass spectroscopy the two major components were in order of their retention time—dibutyl sebacate, and butyl phenyl phthalate: both common plasticizers for PVB. The presence of dibutyl sebacate and butyl phenyl phthalate was confirmed from their GC retention time, their mass fragmentation patterns and the molar ratio was determined by GC to be 24:1. There was a significantly higher amount of dibutyl sebacate than phthalate.

The FT-IR spectra of the hexane soluble extracts after evaporation of the solvent was consistent with that of an ester, with intense bands at 2938, 1736, 1175, and 1142 cm^{-1} and indistinguishable from the spectrum of dibutyl sebacate. There were no strong aromatic bands consistent with butyl phenyl phthalate being present only as a minor component. The IR spectrum of the plasticizer was consistent with it being an aliphatic ester.

The presence of plasticizer in PVB also interfered with the analysis of the acetate groups in PVB from the relative absorbance of the carbonyl ester at 1730 cm^{-1}.

3.2. *Effect of plasticizer on the glass transition*

Un-plasticized PVB had a glass transition temperature, as measured by DSC and correcting for thermal lag [6], of 347 ± 2 K. All commercial PVB samples exhibited glass transition temperatures of 289 ± 2 K, consistent with the presence of plasticizer lowering the value (Table 4). The corresponding value obtained from the recycled waste was 285 ± 2 K. Although these T_g's were not statistically different the increase in the T_g of the recycled material was consistent with a loss of plasticizer.

From Section 3.1 it was concluded that dibutyl sebacate was the dominant plasticizer and it was chosen to investigate the effect of plasticizer concentration on the glass transition temperature, T_g. Cast films were prepared from solutions containing different wt.% plasticizers, these were vacuum dried and their composition determined from the final weight. The reciprocal T_g's increased progressively with dibutyl sebacate content, see Fig. 1, and appeared to obeyed the Fox equation [10], i.e.

$$\frac{1}{T_{g(obs)}} = \frac{T_{g1} + W_1(T_{g2} - T_{g1})}{T_{g1}T_{g2}} \tag{1}$$

where W_1 is the weight fraction of PVB with glass transition $T_{g(obs)}$. T_{g1} and T_{g2} are the glass transitions of PVB and dibutyl sebacate. From the linear relationship the glass transition of dibutyl sebacate was estimated to be 190 ± 20 K. Since for a simple liquid the ratio of T_g/T_m has frequently been observed to be 0.66 the value of T_g was consistent with the observed m.p. (270 K) [11] and a ratio of 0.7 ± 0.1. This plot was used to estimate the amount of plasticizer present in the PVB from the observed value of T_g. Most commercial samples had an average weight fraction of plasticizer of 0.22, which compared favorably with the 15–25% weight loss determined by Soxhlet extraction.

The plasticized samples were also analyzed by FT-IR spectroscopy using an ATR attachment and the absorbance of the carbonyl band at 1730 cm^{-1}. The absorbance increased linearly with plasticizer content; see Fig. 2, obeying the Beer–Lambert relationship, i.e.

$$\log\left(\frac{I_0}{I}\right) = A = \varepsilon c l \tag{2}$$

where I_0 is the incident and I the transmitted beam intensities, A the absorbance, ε the molar extinction

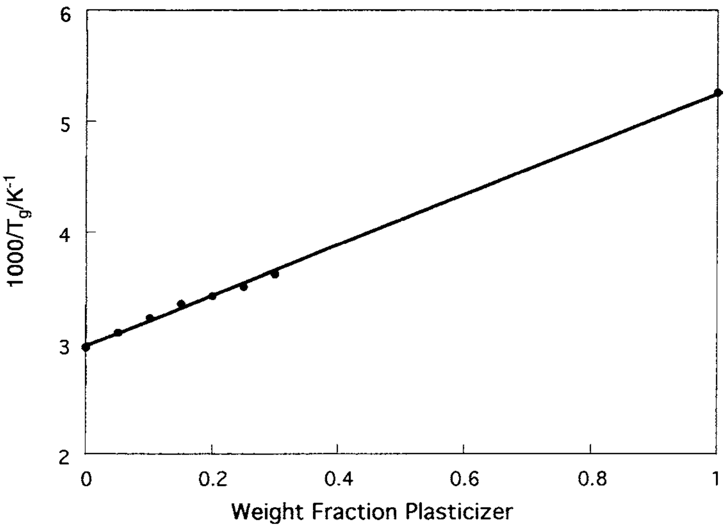


Fig. 1. The dependence of the glass transition of PVB on plasticizer weight fraction, following the Fox equation.

coefficient and *l* the path length of the beam through the sample.

There was a non-zero intercept due to the residual acetate content of the PVB. Using the sebacate calibration as a model this corresponded to about 3.5 ± 1.0 wt.% in reasonable agreement with the NMR estimate of butyral and hydroxyl groups. This was a convenient method also of estimating the plasticizer content of the various PVB samples.

3.3. Thermogravimetric analysis

The relative thermal stability of both recycled and commercial PVB samples were measured by thermogravimetry, from mass loss against temperature plots, see Fig. 3. Weight loss occurred in two distinct regions between 200–300 and 300–500 °C and corresponding to about 20–25 and 70% mass loss. No marked differences were observed between the various commercial

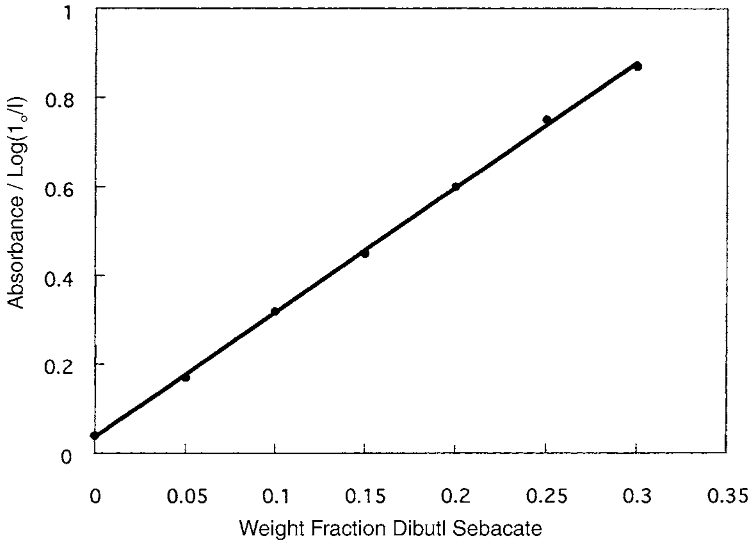


Fig. 2. The variation of the absorbance of the IR carbonyl band with plasticizer weight fraction.

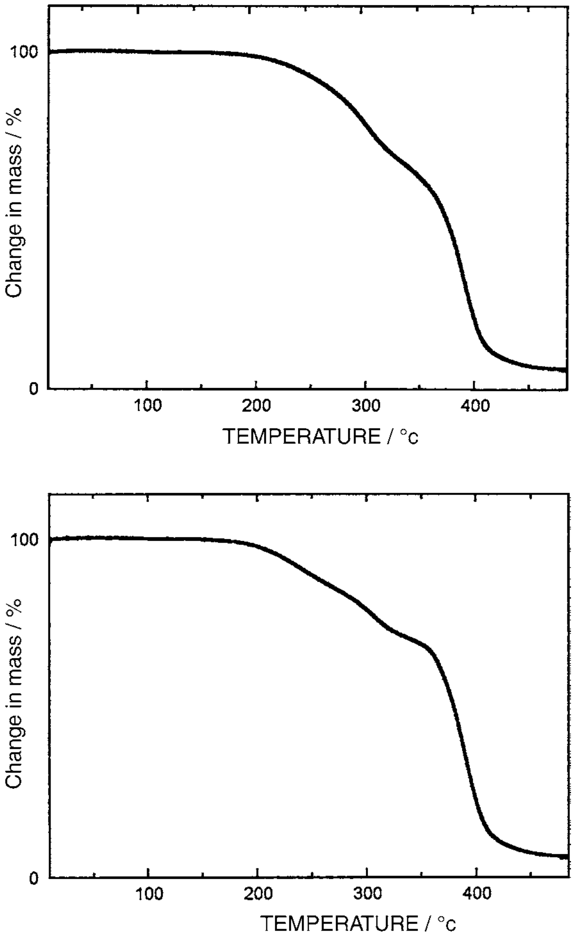


Fig. 3. Thermogravimetric decomposition of PVB. Commercial samples superimposed. Recycled samples superimposed.

grades but the recycled products appeared to lose volatiles at lower temperatures and to give off less, 20 compared with 25%. The final weight loss was the same with all the samples and a brown residue, approximately 5% of the original mass, was left in the crucible.

The evolved volatiles were analyzed by mass spectrometer as a function of time and temperature at fixed *m*/*z* ratios of 59, 69, 71, 77, and 91. These corresponded to the top *m*/*e* ratio for acetic acid, butenal, butyraldehyde, benzene and toluene—the expected products from the thermal decomposition of PVB. Plots of ion current against temperature showed the change in degradation products with temperature see Fig. 4. Little or no degradation products were observed below 250 °C although the PVB samples had lost about 20–25% by mass under these experimental conditions. The major products of the decomposition above 260 °C were initially butyraldehyde and butenal produced by the elimination of butyral groups, followed by acetic acid above 380 °C from the elimination of acetate groups. Acetic acid was a minor component of the volatile degradation products consistent with its content in the original PVB. Aromatic species, such as benzene and toluene, were also observed. These have been attributed to the break down of the polyene products produced by the elimination reactions.

The degradation of PVB samples, from 200 to 250 °C, was also followed by changes in their IR spectra. There was a large reduction in the intensities of the carbon hydrogen stretching and bending modes as well as the carboxyl absorption band at 1730 cm^{-1} but little or no change in bands attributed to butyral groups. These changes were consistent only with the loss of plasticizer.

Up to 260 °C, 25% mass loss was observed over the first 75 min. Isothermal mass loss rate studies were carried out to elucidate the low temperature process, see Table 5. At higher temperatures greater yields of volatiles were obtained and this was attributed to the butyral groups progressively degrading.

From the relative % mass loss and the absence of volatiles detected by the mass spectrometer it was deduced that the PVB was primarily losing plasticizer in the temperature volatilization process between 200 and 260 °C. The loss of additives from a polymer is a complex process involving diffusion, transport and evaporation from the surface of the polymer. Isothermal thermogravimetry has been applied [12,13] to the volatilization of plasticizer from polymers from which it has been deduced that the rate-controlling step is evaporation and the rate of loss is related to the vapor pressure of the plasticizer, p_2, and is linearly dependent on the concentration of plasticizer in the polymer, through a pseudo-first-order rate constant, k, i.e.

$$-\frac{\mathrm{d}w}{\mathrm{d}t} = kp_2 = kp_2^0 x_2 w_2 \qquad (3)$$

Table 5
Isothermal mass loss after 75 min from sample R-1

	Temperature (°C)					
	200	240	250	265	320	350
Mass loss (±1) (%)	15	22	24	25	55	79

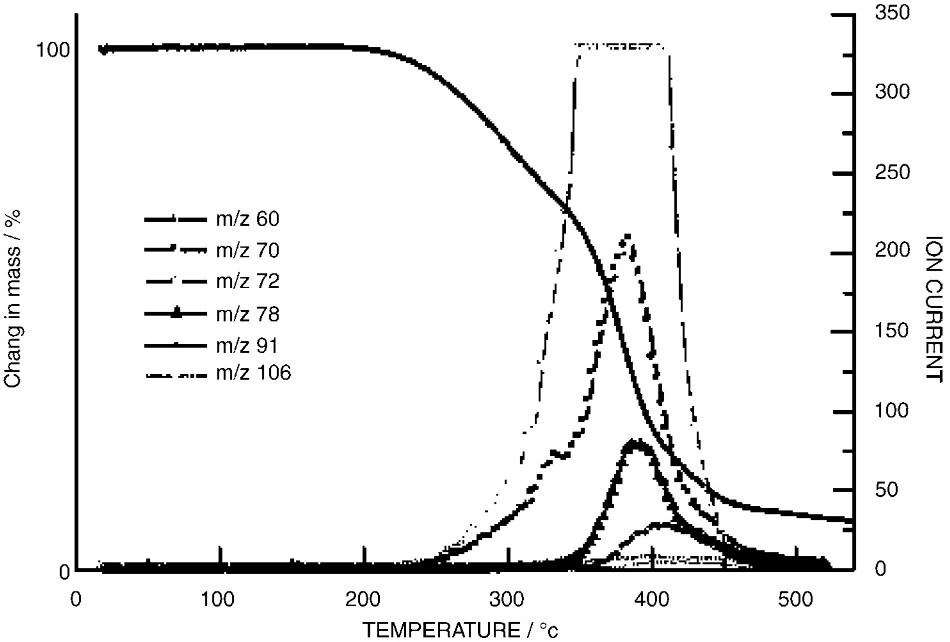


Fig. 4. Thermogravimetric analysis of mixed PVB sample and mass spectrometric analysis of volatiles from PVB with degradation temperature.

The subscript 2 refers to the plasticizer, p^0 the saturated equilibrium vapor pressure, and x the mole fraction.

For p^0 the van't Hoff equation

$$\ln(p^0) = \frac{\Delta S_\mathrm{v}}{R} - \frac{\Delta H_\mathrm{v}}{RT} \tag{4}$$

is valid, where ΔS_v and ΔH_v are the entropy and enthalpy of vaporization of the plasticizer.

The first-order rate constant, kp_2 was determined from the relationship

$$-\ln(1-\alpha) = kt \tag{5}$$

where $\alpha = \Delta W_\mathrm{t}/\Delta W_\infty$, and applied to the fractional weight loss at each temperature as a function of time, t. Plots of $\ln(1-\alpha)$ against t were linear up to about 50% of the weight loss from which an initial rate constant, k_i, was determined. The van't Hoff dependence of the first-order rate constant over 200–250 °C gave a heat of vaporization of 55±10 kJ mol^{-1}.

Adopting Trouton's rule that the entropy of vaporization for a non-polar liquid is about 92 J K^{-1} mol^{-1} the heat of vaporization at the b.p. of dibutyl sebacate, 617 K, [11] was calculated to be 57 kJ mol^{-1}, in sufficient agreement with the above value to suggest that the mass loss analyzed was consistent with plasticizer loss.

Table 6
Tensile properties of PVB samples[a]

Polymer serial number	Strain at break (±0.2)	Tensile strength (±1.0) (MPa)	Fracture energy (±2) (J m^{-3})
S-1	5.4	12.3	25
SR-1	5.3	12.2	21
D-1	4.9	10.1	15
DR-1	5.6	13.7	27
M-1	5.8	16.7	31
MR-1	5.0	13.9	28
50:50 blends			
S-1/D-1	5.3	11.5	23
SR-1/DR-1	4.8	14.2	22
D-1/M-1	5.3	17.0	28
DR-1/MR-1	5.8	14.0	26
S-1/M-1	6.0	13.8	27
SR-1/MR-1	5.5	14.0	28
33:33:33 blends			
M-1/S-1/D-1	5.4	12.7	26
MR-1/SR-1/DR-1	5.7	14.3	30

[a] Average of five determinations.

3.4. The mechanical response of plasticized PVB

The tensile properties of plasticized PVB samples were measured at room temperature on standard dumbbell-shaped specimens cut directly from the films. All the samples were amorphous and above their glass transition at room temperature. The stress–strain behavior was typical of a rubber [15] showing extensive strain hardening, see Fig. 5A and B. However, the strain was totally recovered on removing the stress. Recycled and commercial material had very similar characteristics, see Table 6, in that the strain at break was typically 500–600% in all the samples, and blends, but the recycled blends had higher tensile

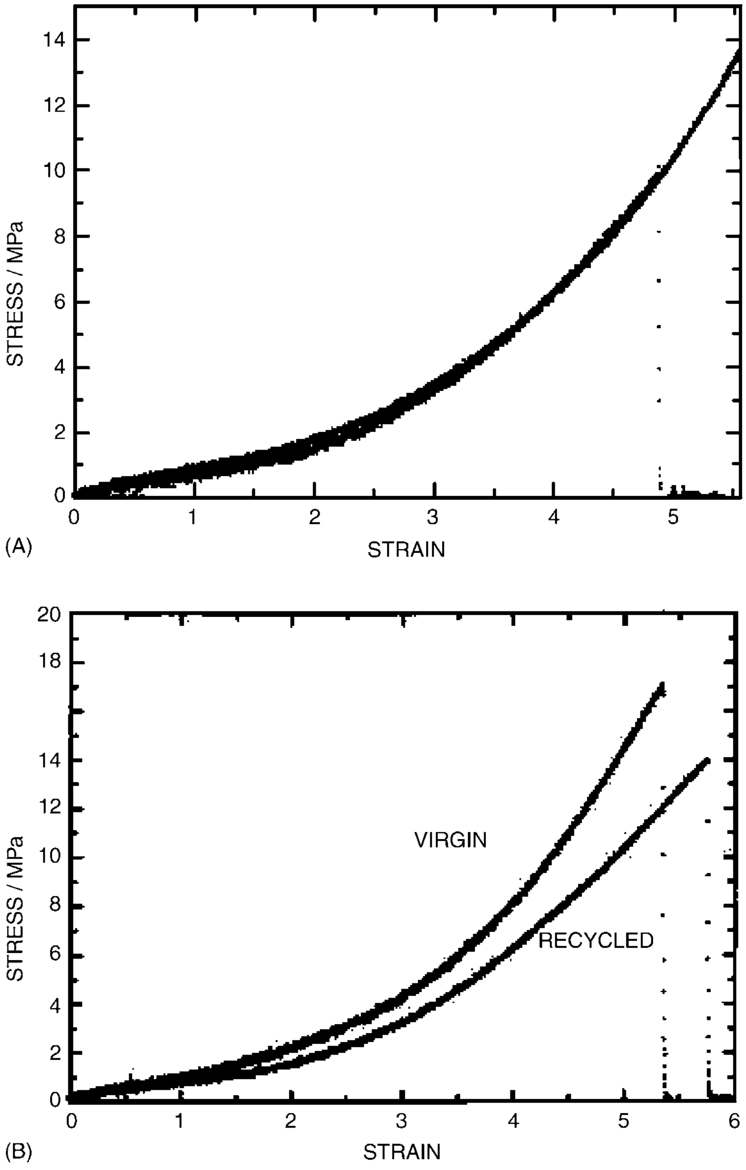


Fig. 5. (A) Stress–strain behavior of PVB samples D-1 and DR-1. (B) Stress–strain behavior of PVB samples D-1/M-1 and DR-1/MR-1 blends. (C) Stress–strain behavior of PVB. Setting of the limits in the Mooney–Rivlin analysis. (D) Mooney–Rivlin plots for PVB samples D-1 and DR-1. (E) Mooney–Rivlin plots for PVB blend samples D-1/M-1 and DR-1/MR-1.

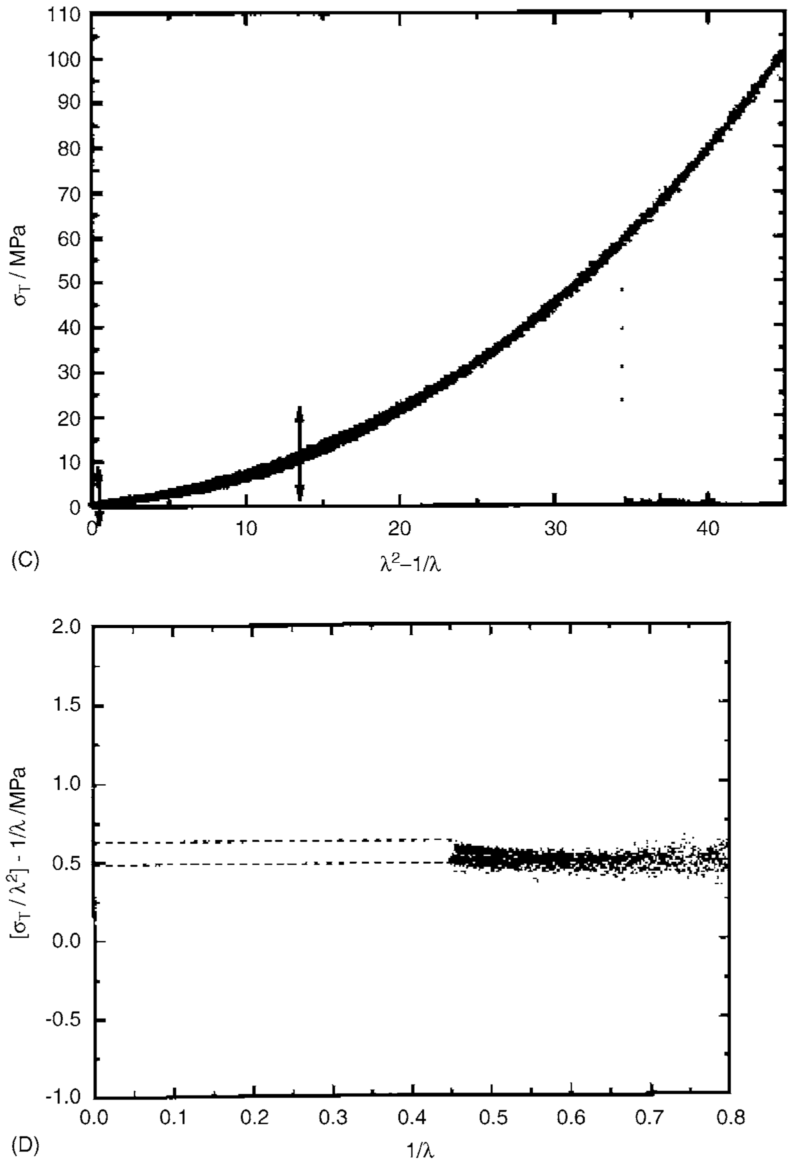


Fig. 5. (*Continued*).

strengths. Clearly, there was no incompatibility between the various grades of PVB or deleterious interactions between the plasticizers present.

The Mooney–Rivlin relationship between tensile stress, σ_T, and strain, λ, was used to analyze this tensile behavior and derive the Young's modulus, G, and entanglement molecular weight, M, as well as the Mooney constants, C_1 and C_2. The relationship was derived from symmetry constraints and consideration of the stored energy within the sample as a function of extent of deformation, λ, [13,14], i.e.

$$\sigma_T = 2C_1\left[\lambda^2 - \frac{1}{\lambda}\right] + 2C_2\left[\lambda - \frac{1}{\lambda^2}\right] \qquad (6)$$

where C_1 and C_2 are the Mooney constants and $\lambda = l/l_0$ with l_0 and l the original and deformed lengths of the specimen.

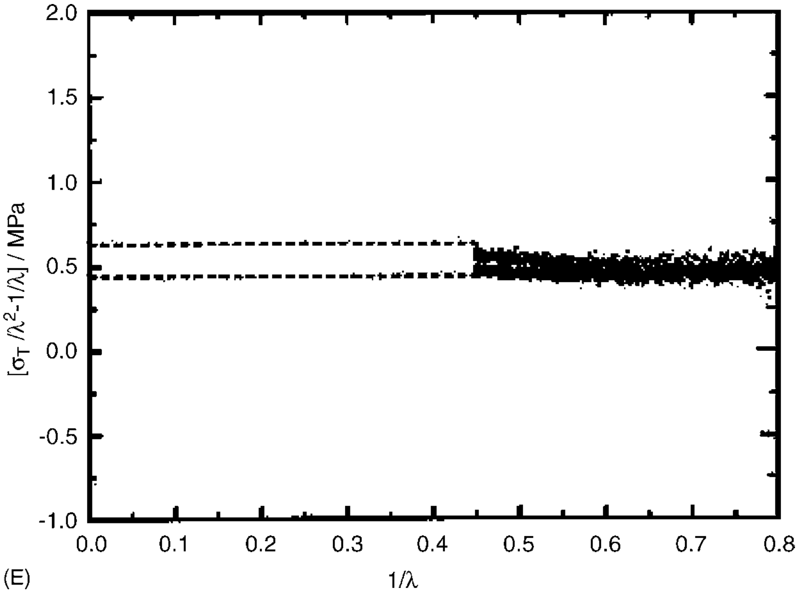


Fig. 5. (*Continued*).

From rubber elasticity theory, the tensile stress is related to extension by the following relationship, i.e.

$$\sigma_T = G\left[\lambda^2 - \frac{1}{\lambda}\right] \tag{7}$$

where G is the Young's modulus. From Eqs. (6) and (7)

$$\frac{\sigma_T}{\lambda^2 - 1/\lambda} = G = 2C_1 + \frac{2C_2}{\lambda} \tag{8}$$

$$G = 2C_1 + 2C_2 \tag{9}$$

Table 7
Rivlin–Mooney analysis of tensile behavior of the PVB samples[a]

Polymer serial number	Modulus G (MPa ± 0.03)	Number of chains per unit volume ($\times 10^{-26}$ m^{-3})	Entanglement molecular weight (kg mol^{-1})	C_1 (MPa)	C_2 (MPa)
S-1	0.49	1.19	5.36	0.16	0.09
SR-1	0.46	1.12	5.69	0.26	−0.03
D-1	0.48	1.17	5.45	0.32	−0.08
DR-1	0.46	1.12	5.69	0.25	−0.02
M-1	0.38	0.92	6.90	0.33	−0.14
MR-1	0.40	0.97	6.56	0.36	−0.16
50:50 blends					
S-1/D-1	0.77	1.87	5.41	0.11	0.28
SR-1/DR-1	0.47	1.14	5.59	0.31	−0.08
D-1/M-1	0.42	1.02	6.25	0.31	−0.11
DR-1/MR-1	0.45	1.07	5.96	0.22	−0.01
S-1/M-1	0.57	1.39	4.59	0.16	0.13
SR-1/MR-1	0.63	1.53	4.17	0.22	0.10
33:33:33 blends					
M-1/S-1/D-1	0.76	1.85	5.45	0.16	0.22
MR-1/SR-1/DR-1	0.44	1.07	5.96	0.25	−0.03

[a] Average of five determinations.

These equations are only valid at small strains and the stress–strain behavior was analyzed, from plots of $\sigma_T/[\lambda^2 - 1/\lambda]$ against $1/\lambda$ only from $\lambda = 1.0–2.5$, see Fig. 5D. This is consistent with the extended chains retaining a Gaussian distribution between chain segments that is inherent in the rubber elasticity theory adopted in this derivation. A constant dependence was observed for commercial, recycled samples and their blends, see Fig. 5D and E, from which $2C_1$, $2C_2$ and G were determined. There was considerable agreement between these values and no marked differences between all the grades, recycled material or their blends. These values are listed in Table 7 for the various PVB samples.

In all the samples the modulus was extremely low 0.46 ± 0.08 MPa and did not vary substantially between grades. Blending material obtained from different manufacturers did, in general, increase the modulus over that of the parent materials, to 0.60 ± 0.18 particularly the S-1 blends, but this may be attributed to loss of plasticizer in the blending procedure.

4. Conclusions

Thermal analysis has been extensively used to characterize the PVB obtained from a wide range of sources. In general the techniques compliment one another and give a broader insight into the molecular composition of the ter-polymer, the nature and concentration of the plasticizer and their effect on mechanical properties. On balance it would appear that recycled material did not vary substantially from that of the original polymers in that they had similar chemical composition and weight fraction of plasticizer. The materials could be blended together, both commercial and recycled, and no incompatibilities between the various grades were observed. Indeed, the tensile properties were extremely similar. Problems might arise from the loss of plasticizer from the recycled material depending on the processing conditions chosen but the content can consistently be measured by several techniques, variation in the T_g, IR analysis of the carbonyl content or mass loss measurements below 250 °C: adding dibutyl sebacate can then make up the deficiency.

The similarity in chemical composition, plasticizer content and mechanical behavior as a soft highly extensive rubber suggested that the blends and recycled polymer could be used as substitute materials in glass lamination. Sheets of M-1/S-1/D-1 and MR-1/SR-1/DR-1 were prepared and placed between two thoroughly cleaned, washed, and dried glass plates. The laminates were placed in a nip roller to remove air between the glass and PVB interface, and seal the edges between the laminates. The laminates were placed in an autoclave at 45 min at 130 °C and ca. 10.3 bar and then slowly cooled to room temperature. Both laminates had similar optical clarity comparable to commercial laminated glass. It was apparent that the mixed and the mixed recycled PVB grades could be used to laminate without loss of optical clarity.

Acknowledgements

The authors wish to thank Mr. Frank Biddlestone for his technical support and assistance and AKD acknowledges receipt of an EPSRC studentship during the tenure of this work. We are grateful to Richardsons Ltd. for the gift of PVB samples.

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