

## **Modification of polyvinyl butyral rheological properties via blending with ionomeric polyvinyl butyral**

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### SUMMARY

Polyvinyl butyral containing ionomer groups (IPVB) can be blended with conventional polyvinyl butyral (PVB) in order to modify rheological properties of the latter including ambient temperature stiffness and flow at processing temperatures. Thus by blending IPVB with PVB at levels up to 50 percent by weight, increases in the ambient temperature stiffness as measured by the storage modulus of the polymer blends ( $G'$ ) are achieved while maintaining or improving flow characteristics at processing temperatures.

### INTRODUCTION

Ionomers are a class of ionic polymers consisting of a hydrocarbon backbone and pendant acid groups which are partially or completely neutralized with metal or quaternary ammonium ions (1-3). Typical concentrations of the ionic groups range between 1 and 10 mole percent. The ionic groups by virtue of their ionic bonds enable the polymer to behave as a thermally reversible crosslinked thermoplastic, i.e., at ambient temperatures the ionic bonds impart additional stiffness to the polymer via association, while at higher temperatures the ionic associations are lost thus allowing the polymer to flow (4-7). Therefore by introducing ionomer groups into a polymer, one could modify the rheological properties of the same.

Polyvinyl butyral (PVB) is conventionally used in safety glazings as a plasticized interlayer. The polymer is prepared by acetalization of polyvinyl alcohol (PVOH) and butyraldehyde (BA) and the reaction conditions are carefully controlled to provide the proper balance of residual hydroxyl groups and 1,3-dioxane rings necessary to impart the end use properties (8).

The aim of this work was to prepare ionomers of polyvinyl butyral (IPVB) and blend the same at levels up to 50 percent by weight with conventional PVB in order to change the rheological properties of the latter.

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## EXPERIMENTAL

### Synthesis:

IPVB samples were synthesized by initially reacting the sodium salt of o-benzaldehyde sulfonic acid (BSNA) with a solution of PVOH in de-ionized (DI) water using nitric acid catalyst at 15°C for 2.0 hours. Next, BA was added to the system and after the resulting polymer precipitated, the reaction was allowed to continue for another 4.0 hours at the end of which the contents were neutralized with sodium hydroxide, filtered and dried. The PVOH used was a low molecular weight material (DP=550).

A sample of standard Monsanto Chemical Company PVB made from a PVOH with a DP of approximately 1800, was used as the control and for blending with the IPVB.

The IPVB/PVB blends as well as the PVB control were plasticized with di-n-hexyl adipate plasticizer.

### Nuclear Magnetic Resonance Spectroscopy (NMR):

NMR spectra were recorded using a Varian VXR400 NMR spectrometer operating at 400 and 100 MHz proton and carbon frequency, respectively. Spectra were recorded at 50°C using d-4 methanol as the solvent.

### Rheological Analysis:

Dynamic mechanical analysis of the polymers were carried out using a Model 605 Rheometrics Mechanical Spectrometer to measure the storage modulus ( $G'$ ), loss modulus ( $G''$ ) and loss tangent ( $G''/G'$ ) in the shear mode at a frequency of 1 Hz from 20°C to 230°C.

## RESULTS AND DISCUSSIONS

Confirmation of the substitution of the ionomer groups were made from the 1-H and 13-C NMR spectra which showed the presence of aromatic groups in the 1-H spectrum and acetal carbon resonances in addition to the conventional PVB acetals in the 13-C spectrum.

The rheological properties are summarized in Table 1, which compares the storage modulus ( $G'$ ) data for IPVB's containing 3 and 5 mole percent ionomer groups, PVB controls (no ionomer groups), and IPVB's blended with PVB control at 10, 30 and 50 weight percent of the IPVB's. It can be seen that in the case of the IPVB's, the  $G'$  values up to 60°C are higher than those of the PVB controls, with the values increasing with

accompanying increases in the level of IPVB in the blends. Furthermore, the  $G'$  values also increase with an increase in the level of ionomer groups in the polymer (3 versus 5 mole%). The increase in  $G'$  at temperatures up to  $60^\circ\text{C}$  is a direct consequence of the increase in stiffness due to the presence of ionomer groups. At the same time, at higher temperatures, such as  $150^\circ\text{C}$ , the  $G'$  values decrease with an increase in IPVB levels. This is due to the increase in the lower molecular weight IPVB component in the blends as well as the thermal dissociation of the ionomer groups at higher temperatures. The  $G'$  and loss tangent profiles for the unblended IPVB's at 3 and 5 mole% ionomer levels and the PVB control are shown in Figure 1. It can be seen that the loss tangent curves for the IPVB's are broader and lower in intensity than the PVB control. This is due to crosslinking or associations due to the presence of ionomer groups. The  $G'$  curves show that the IPVB's are comparable in stiffness to the control at temperatures below  $60^\circ\text{C}$  and beyond this temperature the IPVB's demonstrate increased flow as seen by their lower  $G'$  values.

## CONCLUSIONS

In conclusion, we have demonstrated that the rheological properties of polyvinyl butyral (PVB) including ambient temperature stiffness and processing temperature flow, can be modified/improved by blending ionomeric polyvinyl butyral (IPVB) with conventional PVB.

TABLE 1. EFFECT OF BLENDING IPVB'S  
WITH PVB ON THE STORAGE MODULUS ( $G'$ )

PVB/IPVB	%ION	$G'(25\text{C})$	$G'(40\text{C})$	$G'(60\text{C})$	$G'(150\text{C})$
100/0	0	4.2E7	5.3E6	4.2E6	7.1E5
90/10	3	5.5E7	6.2E6	4.0E6	5.1E5
70/30	3	6.5E7	6.6E6	4.2E6	3.9E5
50/50	3	8.6E7	8.2E6	3.9E6	2.4E5
0/100	3	4.2E8	1.3E8	6.6E6	1.5E5
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90/10	5	5.4E7	6.7E6	4.4E6	6.1E5
70/30	5	7.7E7	9.2E6	5.1E6	5.3E5
50/50	5	1.3E8	1.6E7	5.5E6	3.9E5
0/100	5	4.9E8	1.3E8	7.5E6	1.6E5

$G'(N/M^2)$ ; %ION IN IPVB

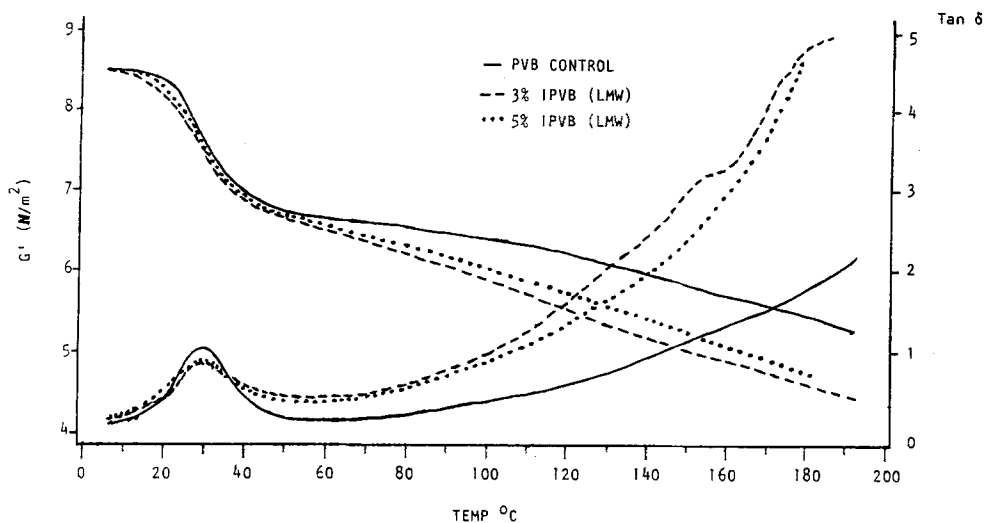


Figure 1. Shear Storage Modulus ( $G'$ ) and Loss Tangent ( $G''/G'$ ) of Ionomeric Polyvinyl Butyral (IPVB) and Polyvinyl Butyral Control (PVB).

### ACKNOWLEDGEMENTS

We would like to express our sincere gratitude to Mr. Cliff Lin and Mr. Robert Rzeszutek for providing the DMA data, Dr's Pierre Berger and Edward Remsen for the NMR analysis.

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