

## Characterization of Polyvinyl Butyral and Sentryglas® Polymers Used as Interlayers in Laminated Glass

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**The polymers used as interlayers in the laminated glass support the tensile stresses, keeping the glass fragments fixed in the film and avoiding possible accidents. This article aims to characterize the polymers Polyvinyl Butyral and Sentryglas®, as their physical-chemical and mechanical properties. It may help a laminated glass company located in Bahia to optimize its production process. We tested the samples to Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA), and Tensile. The results allowed us to characterize the materials technically, comparing them with the data from the sheets provided by their manufacturer. In addition, it has provided the Laminated Glass Factory with data that can reduce costs and improve production. Keywords: Laminated Glass. Polymers. Polyvinyl Butyral. Sentryglas®.**

### Introduction

The glass lamination process consists of two or more layers of glass intercalated with one or more films, which increases its mechanical resistance. In addition, the intermediate layer supports the tensile stresses, preventing the glass fragments from detaching in case of breakage, making it a safety glass. The intercalary material can be PVB (Polyvinyl Butyral), SGA (Sentryglas®), and others [1,2].

PVB is a nonlinear viscoelastic material with deformability and sensitivity to temperature [2]. The reaction of polyvinyl alcohol with butyraldehyde produces PVB, characterized as a random amorphous terpolymer [3,4]. Due to its mechanical properties, PVB is widely used as a protective film, especially in the automotive industry, where vehicle windshields have laminated glass [5].

SGA film is an ionomer that has a structure composed of hydrocarbons with acid groups, commonly obtained by copolymerization of olefins (ethylene, butadiene, or styrene) and functionalized monomers (acrylic acid, p-styrene

sulfonic acid, or methacrylic acid) [6]. The American company DuPont developed this film for the glass facades to resist hurricanes in the United States. Furthermore, due to its mechanical and durability advantages, the film's use expanded in areas where storms do not occur [1]. In Bahia (Brazil), a laminated glass factory provides glass solutions focused on processing monolithic and tempered glass by laminating them to make them resistant and safe glass. To improve the company's productivity, reduce costs, and optimize its process, we studied the properties of PVB and SGA, which are the interlayers used by the company.

The objective is to have better process control because the quality of the laminated glass depends directly on the behavior of the film when it adheres to the glass. In the lamination process, sequences of heating are carried out so that possible delamination does not occur, which is the separation of the glass after the product is ready or imperfections that could prevent the commercialization of the product. In addition, proper conditioning of the PVB, with temperature and humidity control, can have its physical-chemical properties compromised due to its ability to absorb water and thus lose resistance.

For this reason, this study aimed to evaluate the physicochemical and mechanical properties of PVB films with and without conditioning. For comparison, we also achieved the characterization of the SGA film. This work consists of exploratory, experimental research, quantitative and qualitative.

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## Materials and Methods

### Materials

We collected the PVB and SGA films in a lamination industry in Bahia, Brazil.

#### PVB

The Japanese company Kuraray Co., Ltd. produced the PVB films collected. Table 1 shows the properties of PVB film made available from Kuraray.

**Table 1.** PVB properties [7].

Properties	Values
Density (g/cm <sup>3</sup> )	1.065
Tensile strength (MPa)	> 23
Tensile elongation (%)	> 280

We collected six PVB films with dimensions of 280 x 200 x 1.52 mm. Three films were hermetically wrapped in aluminum foil (PVB A) and kept at a controlled temperature of  $17 \pm 5^\circ\text{C}$  and humidity of  $21 \pm 7\%$ . And we kept the other three films in an uncontrolled environment (PVB NA).

#### SGA

The Japanese company Kuraray Co., Ltd. also produced the SGA films collected. Table 2 presents the properties of the SGA film made available by Kuraray.

**Table 2.** SGA properties [8].

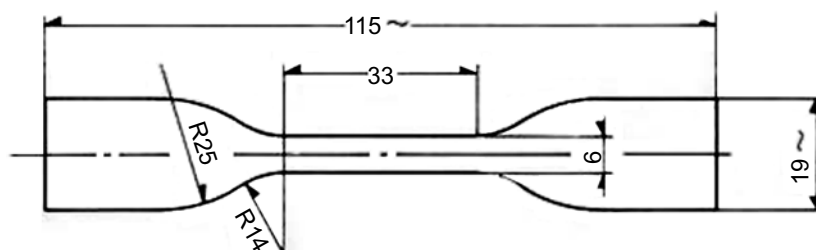
Properties	Values
Density (g/cm <sup>3</sup> )	0.95
Tensile strength (MPa)	34.5
Tensile elongation (%)	400

We collected three SGA films with dimensions of 280 x 200 x 0.76 mm. There was no need to keep the SGA films in an environment with controlled temperature and humidity since these parameters do not affect the physicochemical properties of the material [8,9].

### Methods

We characterized the films by Fourier Transform Infrared Spectroscopy (FTIR) in a Nicolet iS 10 (Thermo Scientific, Massachusetts, USA) with a spectral range of 4000 to 400 cm<sup>-1</sup>, resolution of 4 cm<sup>-1</sup>, and 32 scans. TA Instruments DSC model Q3 analyzer (TA Instruments, New Castle, USA) performed the thermogravimetric (TGA) with a heating rate of 10 °C/min in a nitrogen atmosphere over a temperature range of 25 °C to 700 °C. The uniaxial tensile tests were performed in a universal machine model DL 2000 (EMIC, São Paulo, Brazil) under a constant displacement rate of 50 mm/min, according to ISO 527-1 [10]. For the tensile test, we cut the film in the “tie” model (type IVB), and the ASTM D638 standard determined the dimensions [11] (Figure 1). Ten specimens were used for each type of film to ensure repeatability and a well-defined curve.

**Figure 1.** Tensile specimen dimensions, based on ASTM D638 standard [11].



All dimensions are in mm

## Results and Discussion

### Fourier Transform Infrared Spectroscopy – FTIR

Figure 2 shows the FTIR spectra of PVB A and PVB NA films.

We observed that PVB A and PVB NA showed similar FTIR spectra, corroborating the results of Xiang and colleagues [12]. Table 3 describes the characteristics of each band identified in the FTIR spectra of PVB A and PVB NA.

We verified a slight shift of the band related to the OH group for the PVB NA film ( $3434.41\text{ cm}^{-1}$ ) compared to the spectrum of PVB A ( $3402.54\text{ cm}^{-1}$ ) (Table 3). This displacement may result from water absorption, thus changing the molecular structure of the PVB NA film. We considered that PVB is an amorphous random terpolymer having as a composition the monomers vinyl butyral (76%-80% by weight), which is a substance that does not absorb water and has elastic properties. And vinyl alcohol (18%-22% by weight) and vinyl acetate (1%-2% by weight), substances that can absorb water. In addition to providing better adhesion to inorganic materials such as glass [6,13]. Figure 3 shows the FTIR spectrum for the SGA film.

The spectrum observed for the SGA film (Figure 3) shows characteristic bands of polyethylene and methacrylic acid [14]. Table 4 describes the characteristics of each band identified in the FTIR spectra of SGA.

The FTIR results (Table 4) confirm that there was no change in the chemical properties of the SGA film. With bands characteristic of an ionomer based on poly (ethylene methacrylic acid) [9].

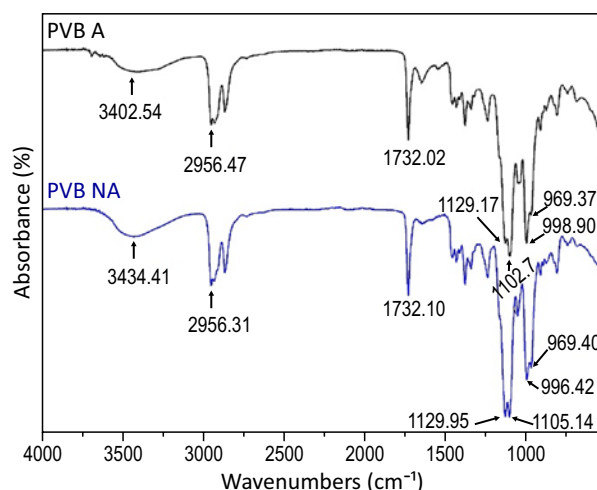
### Thermogravimetric Analysis – TGA

Figure 4 shows the TG and DTG curves of the PVB A film.

Figure 4 shows three mass loss events identified in the DTG curve. Events 1-3 occur at  $215.92^\circ\text{C}$ ,  $294.65^\circ\text{C}$ , and  $388.03^\circ\text{C}$ , respectively. Figure 5 shows the TG and DTG curves of the PVB NA film.

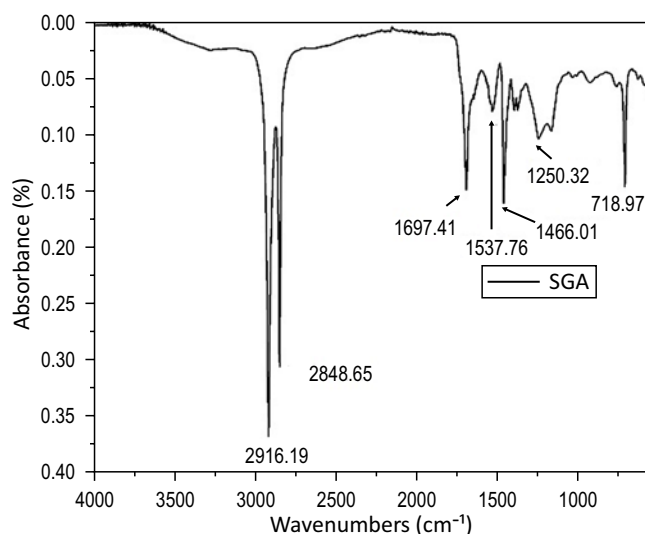
Analyzing the thermal behavior of the PVB NA film (Figure 5), we identify three mass loss events at the DTG curve. The first occurs at  $218.01^\circ\text{C}$ , the second at  $288.18^\circ\text{C}$ , and the third at  $382.17^\circ\text{C}$ . We observed similar thermal behavior when comparing the TGA results of PVB A (Figure 4) and PVB NA' (Figure 5) films. The mass reduction in the first two events for the P films is related to the OH (Hydroxyl) group, with

**Figure 2.** FTIR spectra of PVB A and PVB NA films.



**Table 3.** Description of the FTIR results of PVB A and PVB NA.

Wavenumber (cm <sup>-1</sup> )		Characteristics	Similar Band Material
PVBA	PVB NA		
3402.54	3434.41	Stretching vibration of OH (Hydroxyl)	Vinyl alcohol
2956.47	2956.31	C-H (Hydrocarbon) stretching vibration	-
1732.02	1732.1	Elongation vibration of C=O (Carbonyl)	Acetate vinyl
1129.17	1129.95	Shear bending vibration of the CH <sub>2</sub> (Hydrocarbon) group	Vinyl butyral
1102.77	1105.14	Shear bending vibration of the CH <sub>3</sub> (Methyl) group	Vinyl butyral
998.90	996.42	C-O-C stretching vibration (Ether)	-
969.37	969.4	C-O stretching vibration (Carbon Monoxide)	-

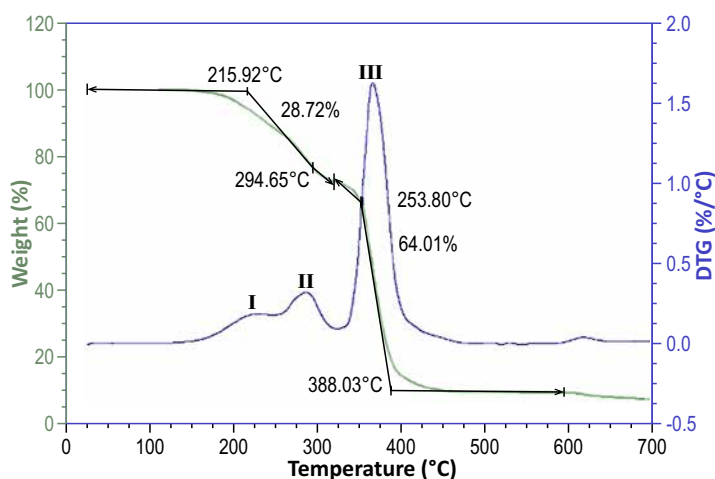
**Figure 3.** FTIR spectrum of the SGA film.

water loss. In the third event, the degradation of the butyric group (C<sub>8</sub>H<sub>14</sub>O<sub>2</sub>)<sub>n</sub> occurs [12]. In the second event, there was a more significant mass loss for the PVB NA film (30.26%), higher than PVB A, with a loss of 28.72%. This higher mass loss of the PVB NA can be justified by water absorption because an environment with controlled humidity cannot store this material.

Figure 6 shows the TG and DTG curves of the SGA film, and we can analyze two mass loss events. The first is at 425.78°C, and the second is at 472.80°C. Because it is a structural film, there was a considerable mass loss of 90.96% above 400°C. According to Yifan and colleagues [15], this mass loss is related to the volatilization of the decomposed polymer chain. Comparing the TG/DTG curves of

**Table 4.** Description of the FTIR Results of the SGA.

Wavenumber (cm <sup>-1</sup> )	Characteristics	Similar Band Material
2916.19	Asymmetric CH <sub>2</sub> stretching absorption (Hydrocarbon)	Polyethylene and methacrylic acid
2848.65	CH <sub>2</sub> symmetric stretching absorption (Hydrocarbon)	Polyethylene and methacrylic acid
1697.41	Symmetric stretching absorption of C=O (Carbonyl) of carboxylic acid	Methacrylic Acid
1537.76	Asymmetric stretching absorption of RCOO <sup>-</sup> (carboxylate group)	Methacrylic Acid
1466.01	Superposition of absorption with asymmetric bending vibration of the CH <sub>3</sub> (Methyl) group and shear bending of CH <sub>2</sub> (Hydrocarbon)	Polyethylene and methacrylic acid
1250.32	O-C(=O)-C (ester) stretching absorption	Methacrylic Acid
718.97	Balance-type bending absorption of CH <sub>2</sub> (Hydrocarbon) groups when linked in a sequence of 4 or more.	Polyethylene and methacrylic acid

**Figure 4.** TG and DTG curves of PVB A film.

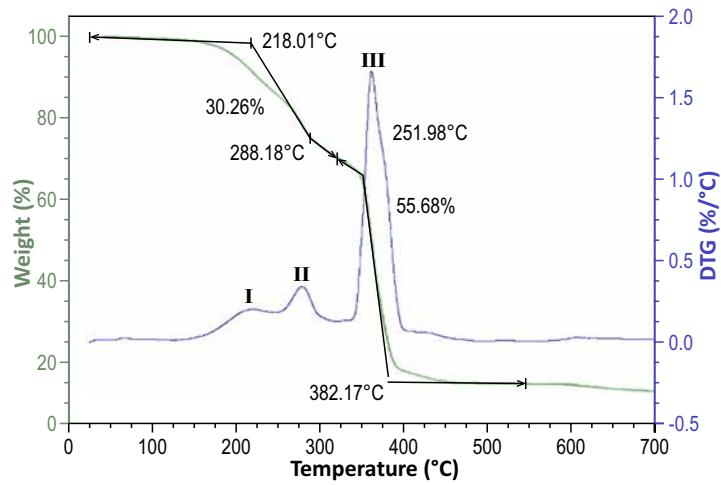
the PVB films and SGA, we can verify that the SGA has a higher thermal resistance, presenting no mass loss in the temperature range of 25°C to 400°C. On the other hand, in this same range, the PVB goes through 3 mass loss events due to the -COONa (carboxylic acid) group providing additional ionic strength to SGA compared to PVB [15,16].

### Tensile Tests

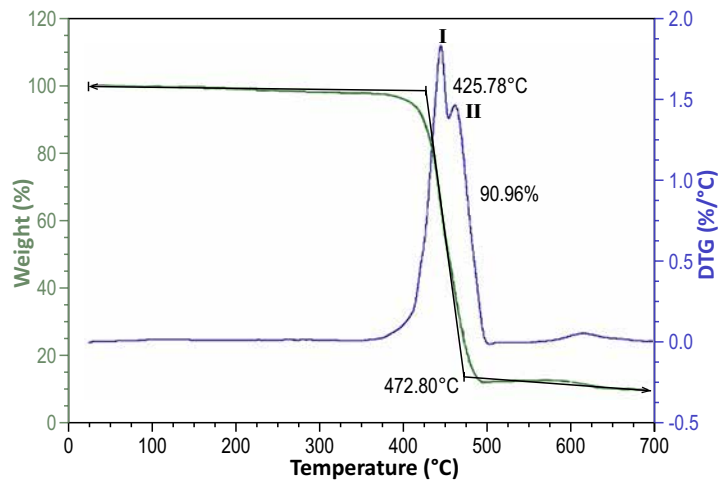
Figure 7 shows the stress-strain curves for PVB A, PVB NA, and SGA films.

The stress-strain curves of PVB films have a small nonlinear elastic area, and a region in which their stiffness gradually increases until rupture

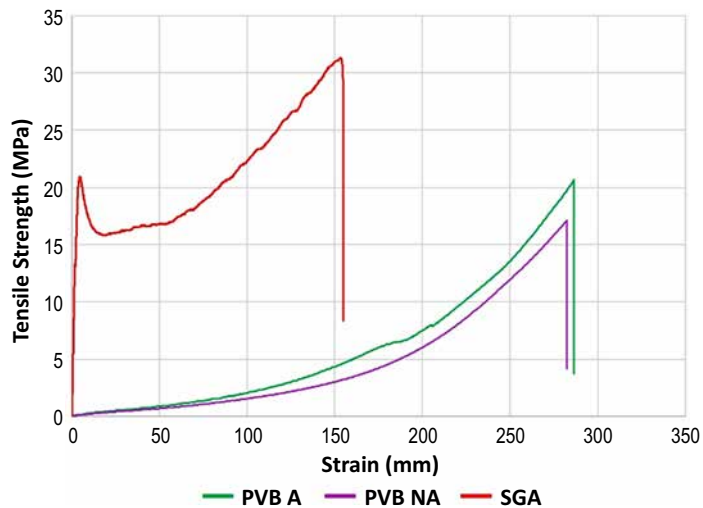
**Figure 5.** TG and DTG curves of PVB NA film.



**Figure 6.** TG and DTG curves of SGA film.



**Figure 7.** Stress-strain curves of PVB A, PVB NA, and SGA films.



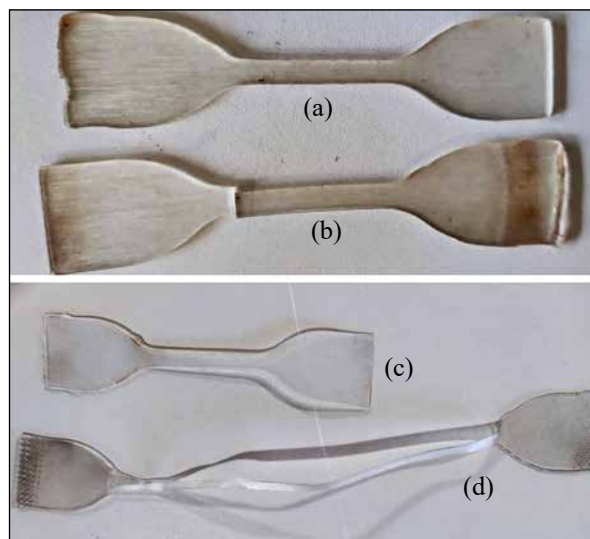
[Figure 8 (b)], characteristic behavior of an elastomer [17,18]. Therefore, PVB is considered a hyperelastic material, in which the stretching of the mesh becomes increasingly tricky as the chains are tractioned, resulting in its stiffening [19]. Besides this, because it is a hygroscopic polymer, it quickly absorbs water, which can interfere with its mechanical properties [6]. The stress-strain curve of the SGA presents a small initial nonlinear region, followed by an elastic zone. Subsequently, a second region with lower stiffness than the initial one characterizes a plastic behavior. Finally, an increase in stiffness until the strain of the specimen (Figure 8 (d)) implies a behavior characteristic of a plastic material [17].

Table 5 shows the uniaxial tensile test data for PVB A, PVB NA, and SGA films.

About the tensile test of films (Table 5), we observed that the PVB A showed maximum stress

of 20.69 MPa, higher than the PVB NA with a pressure of 17.08 MPa. This loss of resistance of the PVB NA film is mainly related to water absorption, occurring in the degradation of the material. The tensile strength of SGA (31.35 MPa) is higher than PVB A and PVB NA, 20.69 and 17.08 MPa, respectively. Because it is a hydrophobic ionomer, it does not absorb water. This polymer is composed of ionic groups, which increases the intermolecular forces between the polymer chains, forming a cross-linked structure and providing greater mechanical tensile strength [8,15,20]. PVB A and PVB NA films had results lower than those stipulated by the manufacturer, which is 23 MPa (Table 1) [7]. The SGA also showed lower tensile strength (31.35 MPa) than that specified by the manufacturer, which is 34.5 MPa (Table 2) [8]. The tensile strength values of the films can vary according to the method applied and the equipment used for the tests.

**Figure 8.** PVB specimens before (a) and after (b) tensile test. And SGA specimens before (c) and after (d) tensile test.



**Table 5.** Tensile test data of PVB A, PVB NA, and SGA films.

Properties	PVB A	PVB NA	SGA
Tensile Strength (MPa)	20.69	17.08	31.35
Deformation (%)	286.89	276.14	158.32

## Conclusions

The FTIR analysis revealed a change in the molecular structure of the PVB films due to the water absorption of the PVB NA film. The SGA showed characteristic bands of the material. PVB A and PVB NA films showed similar thermal behavior, but there was a more significant initial mass loss for PVB NA due to water absorption from the environment. The tensile stress tests showed a decrease in strength for PVB NA compared to PVB A, confirming that water absorption caused the material to lose power. As expected, SGA showed higher tensile stress than PVB films because it is an ionomer. According to the results obtained in this study, it will be possible to establish storage parameters and optimize the production process to reduce production costs.

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