Study of Dicumyl Peroxide in Crosslinking Process of a SBS-Based Pressure Sensitive Adhesive

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Abstract: The styrene-butadiene-styrene (SBS) is a thermoplastic copolymer commonly used as pressure sensitive adhesive (PSA), due its they are highly flexible and elastics, that produce high cohesive strength that results in physical interactions caused by polystyrene (PS) blocks. However, SBS-based PSA's restrain their adhesion properties around their glass transition temperature (Tg), so it is necessary to add a crosslinking agent to increase their thermal resistance. Crosslinking is an important process in adhesives formulations, due that the mechanical properties of adhesive improve, but not enough information I published about the kinetic of that process. One of most studied crosslinking agent for adhesive formulations is dicumyl peroxide (DCP), which shows a good combination of physical and chemical properties to adhesive during crosslinking process. This report presents a crosslinking process using DCP as crosslinking agent. The study of kinetic parameters was determined by differential scanning calorimetry (DSC) in isothermal and dynamic conditions, from which the order of the crosslinking process reaction was found to be first order, according to the Kissinger and Ozawa models. The adhesive and cohesive strength was evaluated through the failure criterion between the bonds of the adhesive line using the single lap join method (SLP), showing that the best performance was that of the formulation made from resin Sylvalite RE100L and 0.81phr of DCP.

Keywords: Adhesives, Crosslinking process, Thermoplastic elastomer.

INTRODUCTION

There are many investigations of pressure-sensitive adhesives (PSA) that use a wide variety of materials from diverse chemical structures, for example: Acrylics, Methacrylic [1], Silicones, block copolymers such as Styrene-Isoprene-Styrene (SIS), Styrene-Butadiene-Styrene (SBS), Styrene-Butadiene-Rubber (SBR) and Styrene-Ethylene-Butylene-Styrene (SEBS) [2, 3]. The development of a PSA consists of a polymer or copolymer and a resin that must be compatible to the polymer blocks, which imparts different viscoelastic properties. Depending on the miscibility of the resin with either copolymer block, the Tg increases; if the resin is compatible with both copolymer blocks, the Tg increases depending on the molecular weight and the Tg of the resin [1, 2]. On the other hand, the compatibility between the tackifier in the SIS, SBS, SBR and SEBS and the intermediate or final blocks of the copolymer, provides them different viscoelasticity properties [3]. In some cases, these block copolymers have a high cohesion strength due to the high glass transition temperature and the association of the external polystyrene blocks in microdomains, regarding the unsaturated central portions (polybutadiene or polyisoprene). This arrangement results in the high elasticity and flexibility of the polymer, and it is the main reason why triblock copolymers like SBS are typically used as thermoplastic base for PSA [4]. However, SBS-based PSA's reduce their operating performance at high temperatures, depending on the Tg of the endblock, in this case, the polystyrene block. These elastomers usually need to be cured to increase their Tg and improve their mechanical properties. Depending on the type of the elastomer, different types of crosslinking processes can be used [5].

The sulfur-based crosslinking method is one of the most popular curing methods, but it just can be partially used in unsaturated polymers. On the other hand, the crosslinking method using peroxide is the second most popular method that generates bonds C-C, which are more stable compared to the sulfur-based methods. Besides, the former method can be used for both saturated and unsaturated polymers. Another advantage of working with peroxide as crosslinking agent is that it does not require heating the material, but it may use UV radiation instead. Such crosslinking method, allows the formation of C-S bonds (bond energy 64 Kcal/mol) and some polysulfide bonds -S_X- (bond energy 34 Kcal/mol), which bond energy is less stable compared to the C-C bond (bond energy 80 Kcal/mol) [6]. Various researches have evaluated the

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changes on the methods like: sample preparation, peel strength of SIS based on hot melt for PSA [2, 7], effect of SIS in PSA mixed with resin (HDCPD). In some investigations, the ethylene vinyl acetate (EVA) copolymer in PSA was studied, while the poly (methyl methacrylate)-b-poly(tert-butylacrylate) (PMMA-b-PtBA) is another copolymer that has been used in PSA formulations [2]. Our research group has carried out the crosslinking evaluation using peroxide as crosslinking agent [3, 6, 8]. Therefore, in this paper, a kinetic evaluation of the crosslinking process of solvent-based adhesive with styrene-butadiene copolymer (SBS) using dicumyl peroxide as crosslinking agent is reported. Kinetic data were obtained from differential scanning calorimetry (DSC), in an isothermal and non-isothermal process.

MATERIALS AND METHODS

Materials

SBS of linear structure with a total content of 33% (SBSH) and 31% (SBSL) of styrene block in the copolymer, was supplied by Dynasol Elastomers (México). Polyterpene styrenated resin commercially identified as Piccolyte®HM106 (R-PICC) (United Kingdom), with a softening temperature of 106°C and a Brookfield (United States) viscosity of 4300 cP at a temperature of 140°C, was supplied by Pinova Inc. (United States). Pentaerythritol ester resin, commercially branded as Sylvalite® RE100L (R-SYL) (United States), with a softening temperature of 100°C and a Brookfield viscosity of 955 cP at 150°C, was supplied by Arizona Chemical Inc® (United States). Dicumyl Peroxide (DCP) was supplied by Retilox® **Co (Brazil)**. The solvents used were n-hexane (99% purity, Sigma), toluene (ACS grade Sigma Aldrich) and acetone (ACS grade Sigma Aldrich) (United States).

Table 1: Formulation of a Standard Adhesive Mixture

Materials	Composition, (phr)
Copolymer	100
Tackifier	40
Dicumyl peroxide	3.6, 2.19, 0.81
Toluene	150
Hexane	150
Acetone	150

Mixing Process

The adhesive formulation was performed in a mechanically stirred glass reactor, using a RW16 IKA Eurostar equipment (Germany). Mixing was developed at room temperature for a period of two hours. A typical formulation, such as that specified in Table **1**, was prepared maintaining a constant amount of R-PICC and R-SYL resins (each separately), but varying the dicumyl peroxide content. The components ratios were established in parts per hundred of rubber (phr).

Adhesive films were prepared by pouring a constant amount of the adhesive blend in a silicone paper container of 10 x 5 x 2 cm, until the solvent was completely evaporated. These films were used for further analysis. The films were cured at 170 °C for a period of two hours, period of time established by previous work conducted by Salazar-Cruz *et al*. [3, 8, 9].

Thermogravimetric Analysis

The adhesive decomposition was studied by thermogravimetric analysis (TGA) using a SDT2960 equipment (TA Instruments) (Delaware, United States) with a heating rate of 10 °C min^{-1} from 40 to 700°C, under nitrogen atmosphere. The sample weight was around 10 mg.

Differential Scanning Calorimetry (DSC)

The thermal properties were measured by means of a *Perkin Elmer DSC8000* (United States) equipment within a temperature interval ranging from 40 to 250 °C; the samples weights were approximately 10 mg. The most commonly used techniques for kinetic evaluation are based on the knowledge of isothermal and non-isothermal processes. In this work, different heating rates: 2, 5, 7, 10, 13, 15, 17 and 20 °C min^{-1} were used for the non-isothermal testing, while for the isothermal tests, the analysis was determined at 170 °C. This temperature was selected due is the DCP decomposition temperature, the objective of this project was evaluate the effect of addition of the crosslinking agent on crosslinking reaction. The isothermal condition was maintained until the thermogram baseline was recovered. The isothermal and non-isothermal methods are widely explained in a previous work [3, 9].

Infrared Spectroscopy

IR analysis was carried out to evaluate crosslinking reaction, by means monitoring typical functional groups of this kind of reaction. IR spectra were recorded with Perkin Elmer Spectrum 100 model spectrophotometer (United States), using for analysis the Attenuated Total Reflectance (ATR) technique, with a diamond/KRS-5 plate, using 12 scans and resolution of 4 cm^{-1}, in a wavenumber range from 4000-400 cm^{-1}.

Adhesive Testing

The adhesive blend was evaluated in wood substrate, the wood substrates were selected from a specific batch of pine wood, which were treated to remove moisture to constant weight and were taken to controlled conditions with relative humidity of 50 ± 2% and at a temperature of 23 ± 1 °C until the equilibrium was reached. The adhesive was impregnated in pine wood substrates with dimensions of 4 x 1 x 0.064 in (Figure **1**), according to the ASTM method D1002-01, applying a wet coating of the adhesive directly onto the pine wood substrates using any type of standard coating equipment. Coating thickness should be adjusted to yield a dry film thickness of 0.5 mm. In this case, it applied the second relase liner, each substrates is direct coated separately with the adhesive. The drying procedure was carried out under controlled conditions (23± 1ºC and 50± 5% RH), waiting 10 min between each layer of adhesive coating. The substrates that show spots, discoloration or any scratches, were discarded. During storage, substrates were under same controlled conditions. Each side of the pine wood substrates is plaed as shown in Figure **1**, four passes of the roller in applying pine wood scpecimen may be made using the nad roller. Unless otherwise specified, conditions the specimens in the specifications described above for not less than 24 hours. For tensile tester, scrolling speed (crosshead) was kept at 0.5 in/min, applying a charge of 10 KN in an equipment, model Instron 3384, evaluating five specimens for each formulation. The objective of this test, was the determination of the strength of adhesion between the substrates of pine wood considering a possible application on lamitates in contact with hot surfaces.

The adherence test was made every 24 hours until the 72 hour, evaluating the takeoff failure load, besides the type of failure present in the union. Adhesive and cohesive failure modes were identified. The cured substrates were prepared in a convection heater model RedLine Binder (Germany) at 170°C.

RESULTS AND DISCUSSIONS

Thermal Stability by TGA

The information obtained by means TGA, was used to determinate the decomposition temperatures of each component in adhesive formulation to find the adequate conditions for crosslinking process. The information about the thermal stability of each component of the adhesive formulation was obtained through a previous study [3], which revealed that the thermal decomposition of DCP begins at 100 °C and its peak temperature (T_p) is set at 170°C, while the T_p of the copolymers is around 450 °C. The initial decomposition temperature represents the point at which the material starts losing weight and is associated to its thermal stability [3, 10, 11]. The decomposition temperature of the R-SYL and R-PICC resins, as well as that of the copolymer (T_p 350°C, 380°C and 450°C respectively) are much higher than that of DCP, which indicates that the selected temperature to evaluate the thermal effect (170°C) of the formulations corresponds to the thermal decomposition of the DCP molecule [3]. It is important to mention that the T_p obtained by TGA, provides us with tools to determine the temperature at which the thermal effect will take place in isothermal conditions.

Evaluation of Kinetic Parameters by DSC

The non-isothermal methods can use either individual temperature sweeps to show the enthalpy change of the reaction, or scanning series with different heating, rates as shown in Figure **2**. This possibility offers advantages due to the reduction of the time needed to carry out the experiments, besides its obvious lost area calculation at the beginning of the reaction, like in the isothermal methods; therefore, the

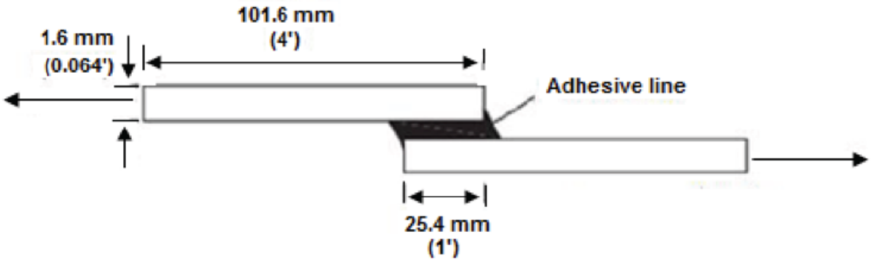

Figure 1: Design of the substrate and the area of the adhesive's impregnation.

last method is more accurate. There are several methods to evaluate the activation energy (E_a) by calculating the E_a parameters based on one or multiple heating rates of the thermogravimetric curves. Some methods based on this principle are Kissinger, Ozawa, Van Krevelen, Coats-Redfern, among others. However, the most widely used methods are the Ozawa and Kissinger equations [3, 10, 12, 13]. This models that used to calculate the activation energy parameters are based on the displacement suffered by peak temperature T_p, as a consequence of modifying the heating ramp (q). The observed shift is a result of the mathematical expression of the kinetic law that was studied by Kissinger and Ozawa, resulting in different methods that can be applied for calculating the activation energy [12]. The displacement of Tp when heating rate change was modified due the changes in the kinetics of crosslinking reaction, however, the apparent activation energy was determinated insingle dynamic curve performed at low and high heating rates, which verifies that there is not significant influence of the heating rate on this phenomenon, indicating that displacement of the Tp is a consequence of the mathematical expression of the kinetic was extensively studied by Kissinger, 1957.

Ozawa Method

According to the Ozawa method, the activation energy satisfies the following equation.

$$E_a = -2.303R\frac{d(\log q)}{d\left[\frac{1}{Tp}\right]} \tag{1}$$

The plot of log (q) versus $1/T_p$ represents a straight line which slope will get the E_a value.

Kissinger Method

In the method proposed by Kissinger, the activation energy is given by the equation.

$$E_a = -2.303R\frac{d\left[\log\left(\frac{q}{T_p^2}\right)\right]}{d\left[\frac{1}{Tp}\right]} \tag{2}$$

The graphical representation of log (q / T_p^2) versus 1 / T_p is a straight line which slope will give the value of Ea. Previous models, especially the Kissinger method, are used to calculate the activation energy of the decomposition and crosslinking processes of the polymers.

These methods cover the determination of kinetic parameters for exothermal reactions and other reactions which behavior can be described by the Arrhenius equation. The determination of the activation energy for the temperature peak requires a very accurate peak identification, as a function of the linear analysis of the heating rate changes. In this method, the sample increases linearly its heating rate, and any exothermal peak is analyzed [12].

The study was focused on the SBS/DCP systems, in concentrations of 0.81 and 3.60 phr of DCP and the corresponding variations of the R-PICC and R-SYL resins, as displayed in Table **1**. In all the adhesive samples, a Tp displacement was observed with increasing heating rate, corresponding to the exothermic behavior of DCP found in the adhesive matrix. Figure **2** shows the thermograms of SBSL-2 / 3.6 phr PDC sample. It is important to notice that this behavior is consistent with that reported before [3, 9, 11-15].

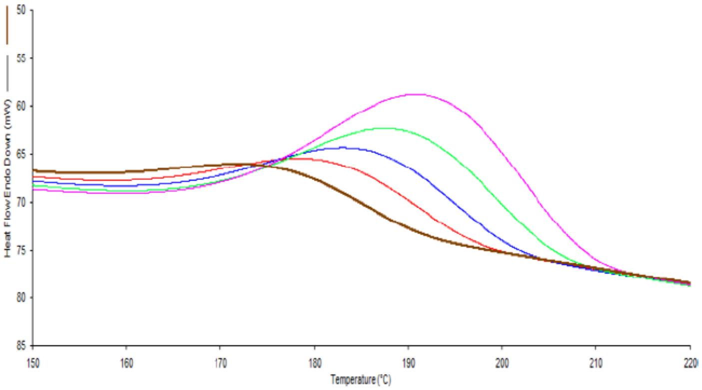

Figure 2: DSC Thermograms showing the influence of the heating rate in Tp, for mixture SBSL-2/3.6 phr PDC.

The enthalpy change is the predominant effect of a structural change, because it triggers an increase in the thermal energy due the epoxide rings rupture, which have significant energetic differences [7]. The amount of heat that is generated, as well as the increasing rate, correspond to the number of the reacting epoxide groups [6].

According to the results, samples result in a sufficiently exothermic process, which allows the study of the crosslinking process kinetics, shown in Figure **2**, where the area under the curve represents the reaction enthalpy of the crosslinking process. This pattern behavior matches with the reported by Vallat *et al.* [16]. In addition, the DCP decomposition is the determining step of the crosslinking process of the adhesive mixture. In this exothermic process, there is a quantitative relationship between the peroxide concentration and the amount of released energy (ΔH), when the exothermic crosslinking process occurs. This behavior was also reported before [3, 9, 11]. On the other hand, the main contribution of this work is the evaluation of the tackifier effect on the adhesive blend. It should be noted that the R-PICC is a styrenated polyterpene resin that is compatible to the polystyrene block and partially compatible to the butadiene block; whereas the chemical structure of R-SYL corresponds to an ester resin, which makes it more compatible to the polybutadiene block than to the rigid one, according to the discussion from previous articles [3, 10, 17].

It is important to observe that in Table **2**, according to the thermal decomposition theory, the E_a values that were calculated via the Kissinger and Ozawa methods are very similar one to each other, and the correlation coefficient R^2 is greater than 0.90, as shown in Figure **3**, which indicates a good linear correlation between the values.

The SBSH and SBSL with DCP/R-SYL systems tend to show lower E_a values than the DCP/R-PICC system, which implies that the crosslinking process is carried out easier in the first systems. The crosslinking

Table 2: E_a Calculations, by Kissinger and Ozawa Methods in the Adhesives Formulation

Sample			Heating Rate						Ozawa		Kissinger	
Code	**Relation**	**(°C/min)**	**5**	**7**	**10**	**13**	**15**	**20**	**E_a(kJ/mol)**	**R^2**	**E_a(kJ/mol)**	**R^2**
SBSH	SYL/3.6 DCP	T_p(°C), peak temperature	155	157	158	167	168	172	106.37	0.91	99.11	0.90
	PICC/3.6 DCP		182	185	189	192	194	197	161.46	0.99	153.77	0.99
	SYL/0.81 DCP		152	152	155	167	172	174	74.82	0.88	67.58	0.86
	PICC/0.81DCP		181	184	184	189	194	197	133.72	0.90	126.04	0.89
SBSL	SYL/3.6 DCP		177	182	185	187	191	194	139.91	0.88	132.28	0.85
	PICC/3.6 DCP		181	185	189	192	194	199	143.36	0.99	135.67	0.98
	SYL/0.81 DCP		163	171	171	177	178	193	77.73	0.98	70.23	0.97
	PICC/0.81DCP		180	181	188	193	194	198	121.53	0.97	113.86	0.97

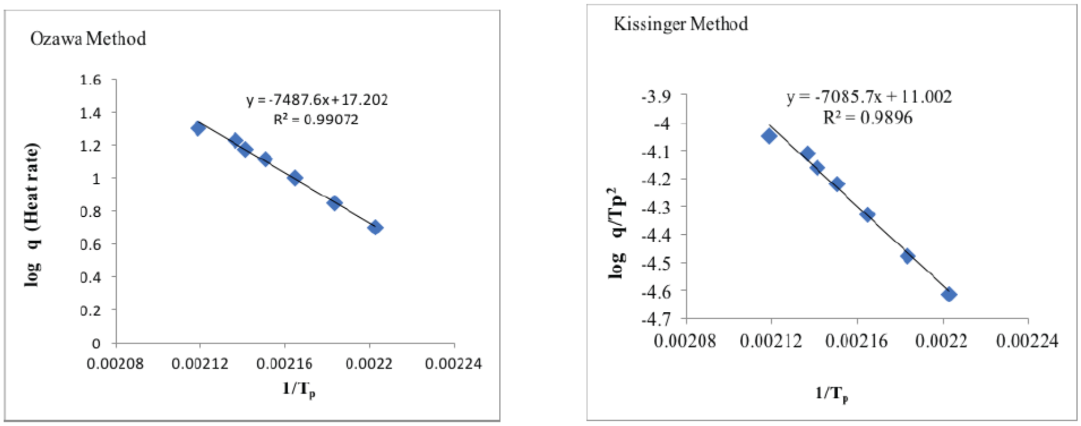

Figure 3: E_a linear regression calculation for the Ozawa and Kissinger methods in the adhesive formulation SBSL/3.60PDC/ R-PICC.

process starts when the DCP molecule breaks down, at the T_p that was determined by TGA, generating peroxyl radicals, as represented in Figure **4**. These radicals are also able to produce methyl radicals that have sufficient energy to abstract a hydrogen atom from the double bonds of the PB unit; they principally attract allyl hydrogens corresponding to the 966 and 724 cm^{-1} signals of double bonds $\gamma(=C-H)\,trans\ y\ \gamma(=C-H)\,cis$ throughout the polymeric chain. Thus, macro radicals are generated, and their combination produces C-C bonds that are responsible for the crosslinking process. Then, the most important signals associated to the crosslinking process appear at 3400 and 1724 cm^{-1} for hydroxyl and carbonyl groups, respectively [3, 6, 9, 11].

The kinetic parameters like the reaction rate constant, **K**, and the reaction order, **n,** were calculated by way of a DSC analysis under isothermal conditions at different temperatures (170, 180, and 190°C). The experimental temperatures were determined by TGA, as reported in a previous work [3]. That study revealed that the best operating temperature for the polymer/resin/peroxide systems, is 170°C, because lower temperatures led to long isothermal time periods, broad and low intense exothermic peaks, and in some occasions, it could lead to inadequate signal/noise relations [3, 9, 11]. Table **3** exhibits the reaction order values for the whole experimental matrix, concluding that the crosslinking process of SBS-based adhesive blends was a first order reaction. The same behavior for similar systems has already been reported by other research groups [6, 9, 11]. From Table **3**, it is evident that the reaction rate constant is strongly influenced by the characteristics of the adhesive mixture; for example, when the R-SYL tackifier is added to the adhesive mixtures, a substantial increase of K takes place, which indicates a higher velocity in the crosslinking process in comparison to the adhesive mixtures containing R-PICC resin. It is important to mention that the linear correlation coefficient that was calculated for the kinetic parameters, was above R^2=0.94.

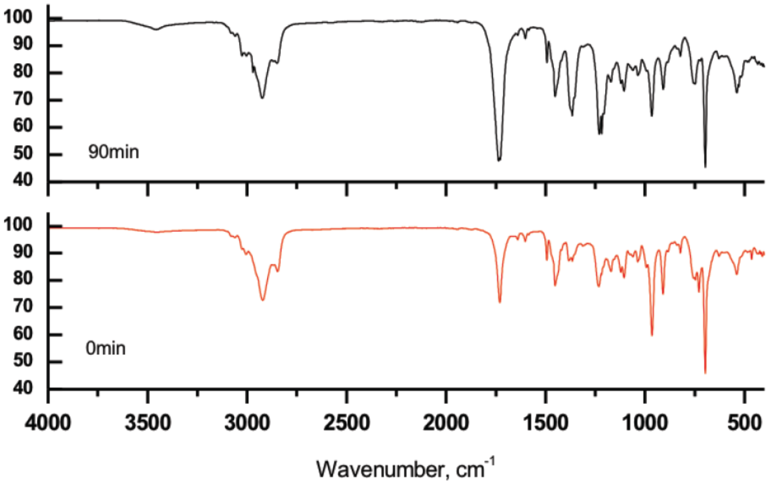

Figure 4: FTIR spectra of thermal decomposition of DCP, **A**) chemical structure, **B**) SBS-H)/R-SYL/3.60phr PDC at 90min., **C**) SBS-H)/R-SYL/3.60phr PDC at 0 min. evaluated at 170°C.

Table 3: Kinetic Parameters Obtained from Isothermal DSC Analysis

Sample		T (°C)	n	ln (K en min^{-1})	R^2
SBSH	R-SYL/3.6 DCP	170	1.0158	1.7231	0.9935
	R-PICC/3.6 DCP	170	0.9046	0.2246	0.9988
	R-SYL/0.81 DCP	170	1.0512	1.7230	0.9409
	R-PICC/0.81 DCP	170	1.0000	0.0632	0.9990
SBSL	R-SYL/3.6 DCP	170	1.1865	0.2774	0.9991
	R-PICC/3.6 DCP	170	1.0217	0.1971	0.9960
	R-SYL/0.81 DCP	170	1.0338	0.1402	0.9858
	R-PICC/0.81 DCP	170	0.9030	0.1017	0.9928

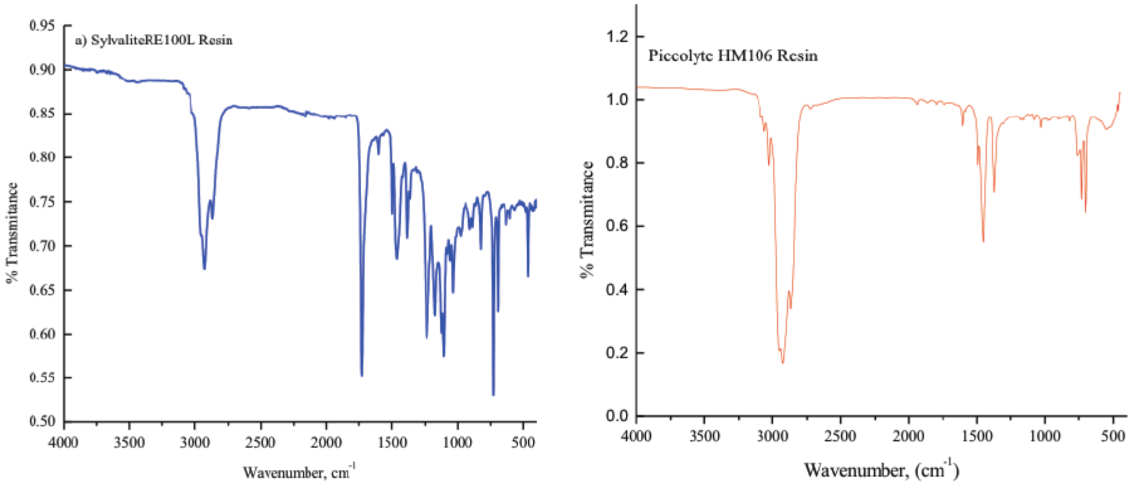

Figure 5: FTIR spectra of SylvaliteRE100L resine a), and PiccolyteHM106 resine.

Figure **5a**) shows the R-SYL infrared spectrum, where signals at 3000 and 1755 cm^{-1} due to unsaturations and the ester group, can be detected. The compatibility between the ester group and the polybutadiene block (PB), produces a structural effect that promotes the constant K raising, as shown in Table **3** for adhesive mixtures with R-SYL; and therefore, it favors the crosslinking process. By contrast, mixtures with R-PICC resin have lower values of constant K due to the miscibility between the polystyrenic block shown in Figure **5b**), which are identified by the methylene group signals at 2495 cm^{-1}, and the overtone signals appearing around 2000-1750 cm^{-1} attributed to aromatic groups that act as a support of the polystyrenic block.

Evaluation of the Adhesive Test

The adhesive unions analysis is important to understand the failure mode and the highest union strength. The specimens were analyzed, until failure, by means of applying a uniaxial tension on them. The experiment consisted in determining the load that was needed to detach the union, in order to identify the failure mode. Table **4** shows the failures on surface of the substrates before curing, and as it can be seen, the surface failures exhibit significant differences based on the type of resin used in the formulation. The adhesive blends prepared from R-PICC resin show an adhesive failure in most of the substrates, while the adhesive mixtures made with R-SYL resin show a failure inside the adhesive layer (cohesive failure) in almost all the substrates. The highest failure load was 738 Kgf/cm^2

Table 4: Failure Shown on the Substrates After the Peel off Test of the Adhesive Union

Material	Process	Resin	Image	Failure
SBS-H	Without Crosslinking	R-PICC		adhesive
		R-SYL		adhesive
	Crosslinked	R-PICC		cohesive
		R-SYL		Cohesive

for the SBSL/R-SYL adhesive blend, as shown in Figure **6**. Table **4** shows a representation of each tackifier-overlapping joint cured at 170°C, revealing that the predominant failure occurs in the adhesive layer (cohesive failure), which takes place in the center of the union.

Table **5** presents the effect of the crosslinking agent on the adhesive force of the cured substrates, and shows that the highest failure load needed to peel off the substrates was 2530.8 Kgf/cm^2 for the SBSL/R-SYL/0.81phr DCP tackifier resin. This behavior is observed because the R-SYL resin is more compatible to the PB block than to the PS block [17], and thus, the crosslinking process is enhanced. The results presented in Table **5** also suggest that increasing the crosslinking agent (DCP) decreases the adhesive force between the substrates, which can be attributed to the rigidity of the PS blocks, because achieving a more rigid adhesive reduces its adhesion properties. Furthermore, this behavior is also triggered by the fact that R-PICC resin has higher compatibility to the PS block than to the PB block.

CONCLUSIONS

As a result of the experimental evaluation that was discussed in this investigation, it can be concluded that the crosslinking process is improved by the addition of low DCP ratios, and by the effect of the R-SYL tackifier, due to its miscibility towards the polybutadiene block; this was determined by the kinetic study that was performed through the Kissinger and Ozawa models, in addition to the performance tests based on the ASTM D1002-01 method. The activation energy was calculated because it represents the amount of energy required to accomplish the crosslinking process. In the activation energy calculation, it was found that the correlation coefficient (R^2) was greater than 0.98 for the SBSL/R-SYL/0.81 phr DCP formulation, obtaining an Ea of 77.73 KJ/mol by using the Ozawa method, and 70.23 KJ/mol via the Kissinger method. The SLP test

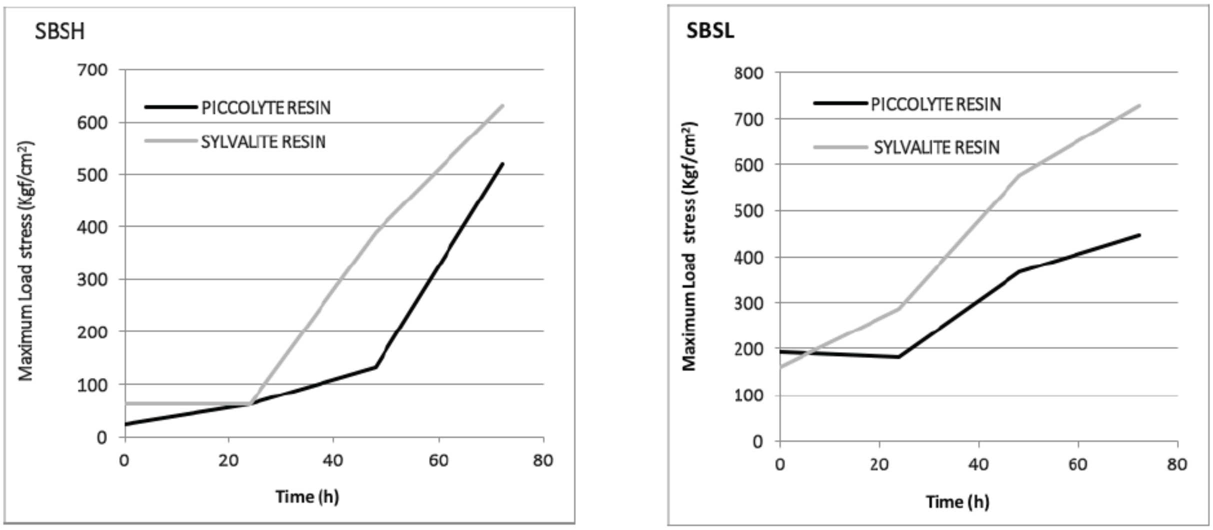

Figure 6: Maximum load stress needed to peel off the adhesive unions, as function of the time.

Table 5: Maximum Load Stress Needed to Peel off the Adhesives Evaluated at 170°C

Adhesive Mixure		Load Average (Kgf/cm^2)			Failure		
Copolymer	Tackifier	phr DCP			Phr DCP		
		0.81	2.19	3.6	0.81	2.19	3.6
SBS-H	R-SYL	2094.2	782.9	296.2	cohesive	cohesive	cohesive
	R-PICC	1927.7	566.6	437.4	cohesive	cohesive	cohesive
SBS-L	R-SYL	2530.8	1900.0	732.8	cohesive	cohesive	cohesive
	R-PICC	1649.2	1055.7	895.0	cohesive	cohesive	cohesive

evaluation of the cured and non-cured specimens confirmed the behavior that was found through the kinetic analysis. It was observed that the crosslinking experiments increased the failure load in more than 300%, indicating a superior performance in the cured PSA.

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