

# Determination of elastomer content in NR/SBR/BR blends

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## Abstract

Determining elastomer content in ternary rubbers via infrared (IR) spectroscopy presents challenges due to spectral band overlap. Coupled techniques could be a solution, but in certain cases it can also involve overlapping events resulting in greater errors. The best option is to use an instrumental technique with appropriate conditions that avoid overlapping. This paper presents the development of transmission/reflection IR methodologies for the content determination of a blend with natural rubber, styrene-butadiene copolymer, and polybutadiene (NR/SBR/BR), with  $A_{887}$  and  $A_{1375}$  bands for NR and  $A_{699}$  for SBR. The BR content, which was calculated by subtraction of the NR and SBR results from the IR methodology, is confirmed by acid-resistance quantitative data. The methodologies errors (2 to 6%), with non-overlapping bands, encompass what is found in the literature (5%). Such development ensures the determination of elastomer content in ternary rubbers with fast and accurate methodologies.

**Keywords:** *acid-resistance, IR, reflection, ternary rubber, transmission.*

**Data Availability:** *Research data is available upon request from the corresponding author.*

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## 1. Introduction

Rubbers are materials extremely important used in the aerospace sector in thermal protections and flexible joints of rocket engines, and in the aeronautical and automotive sectors in tires<sup>[1-9]</sup>. However, especially in the automotive industry, one single type of rubber does not provide all the desired properties in a product. The blending of different types of rubber offers specific performance results for new technological solutions<sup>[10,11]</sup>. Optimum properties can be achieved by controlling morphology, composition, and processing conditions<sup>[12,13]</sup>.

The NR/SBR/BR ternary rubber blend (natural rubber/styrene-butadiene copolymer/polybutadiene) is considered unique, as it has three different binary pairs (NR/SBR, NR/BR and SBR/BR), and that SBR contains BR as a constituent. Consequently, the determination of their rubber content may be complex because infrared (IR) spectra present bands overlapped<sup>[14,15]</sup>. The SBR rubber is the synthetic elastomer with the largest production in the world. BR, with the second largest volume of synthetic rubber produced, is generally mixed with other elastomers such as NR or SBR, and is often the main component in synthetic rubbers<sup>[16-19]</sup>. Recent studies of the NR/SBR/BR blend have focused on analyzing tires, checking their morphology, thermal degradation, and their effect on the environment<sup>[20-22]</sup>. Stoček et al.<sup>[23]</sup> investigated

the cut and chip (CC) resistant of NR/SBR blend (50:50), NR and SBR. The results explained the empirical preference for blends containing NR for improving CC strength.

Adding fillers to a rubber compound modifies its properties<sup>[24,25]</sup>. In relation to this, Chen et al.<sup>[26]</sup> evaluated the addition of nanometer silica and micron kaolin in NR/SBR/BR using IR spectroscopy (qualitatively) by diffuse reflectance (DRIFT) and attenuated total reflection (ATR). The structure and interfacial interactions of the composites were investigated. The study concluded that the coupling agents were mainly coated on silica particles rather than kaolinite surface due to the latter reactive sites being limited and covered by silica particles.

Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) are useful for studying the morphological phases and filler dispersion of rubber blends<sup>[27]</sup>. Jovanović et al.<sup>[13]</sup> analyzed the NR/BR/SBR blend to synthesize and characterize the blend (25/25/50) reinforced with different silica nanoparticles levels (0-100 phr). The IR/ATR analysis was applied qualitatively, and the band shifting from 1450  $\text{cm}^{-1}$  to 1480  $\text{cm}^{-1}$  confirmed the interaction between the blend and the nano-silica. A good interaction between the polymeric

matrix and silica nanoparticles was also demonstrated. In a subsequent study, Jovanović et al.<sup>[28]</sup> employed the same methodology with the addition of carbon black (60 phr) instead of silica. The blend components influence the physical and chemical stability of the final material.

As can be seen in the previously discussed studies, IR spectroscopy has been used mainly qualitatively. Comparatively, quantitative IR studies are fewer in number. Although binary blends quantification has been successfully investigated in the last decade<sup>[29-40]</sup>. In most cases coupling techniques are used for evaluating ternary rubbers, as IR spectra can display overlapping of elastomers characteristic bands<sup>[14]</sup>. Nevertheless, if the mixture components are known, a model with a calibration curve can be developed for content determination.

For many years, the IR/transmission/controlled pyrolysis methodology<sup>[41-43]</sup> has stood out as the only available approach for the analysis of many types of rubber. Harms<sup>[43]</sup> evaluated quantitative composition of diverse elastomers by using calibration curves obtained from thermogravimetric peak heights and from IR bands. Results showed that, as long as the pyrolysis process is controlled, IR data are similar to those obtained by TGA. Pyrolysis techniques<sup>[44,45]</sup> have also been used in IR reflection analysis by different numbers of reflections (multiple reflection-ATR and by universal attenuated total reflectance-UATR). Reflection techniques are useful in cases in which occur polymer and additives bands overlap.

Ghebremeskel and Shield<sup>[46]</sup> applied IR analysis to investigate the composition of SBR, acrylonitrile-butadiene rubber (NBR) and polyvinyl chloride (PVC) ternary blends. IR spectroscopy was used qualitatively and quantitatively for the composition determination of binary and ternary blends. Blends were characterized by ATR without sample preparation. SBR (styrene at 1602 cm<sup>-1</sup>) and NBR (acrylonitrile at 2237 cm<sup>-1</sup>) compositions were independently determined by MIR analysis with the use of two calibration curves. These curves were normalized to predict the SBR/NBR blend ratio. Results evidenced that the PVC presence in the mixture did not interfere with the blend characterization. Test samples were used to determine the methodology accuracy (error ~ 6%).

The NR/SBR/BR content determination is conducted by applying IR/ATR (germanium crystal) and TGA. Bands at 1375 cm<sup>-1</sup> (NR - methyl group), 699 cm<sup>-1</sup> (SBR - aromatic CH group), and 738 cm<sup>-1</sup> (BR - cis C=C group) are selected, along with their intensity ratios. Band height correction is performed using an algorithm<sup>[47]</sup>. The authors conclude that a combined methodology (FT-IR/TGA) is sufficient to quantify polymer compositions of ternary blends of NR/SBR/BR.

Even though the investigation by Datta et al.<sup>[47]</sup> has shown adequate results for the determination of NR/SBR/BR, it utilized two instrumental techniques, which increases complexity, time of analysis and costs. This fact opens a window of opportunity for the development of new methodologies that use solely one instrumental technique, or a simple laboratory test to validate results, such as the methodology presented in this current study. Furthermore, conditions can be modified in the same instrumental technique to obtain different results. In the case of FT-IR, the way of obtaining spectra can be varied, as there are differences in optical path between reflection and transmission spectra<sup>[48]</sup>. This may interfere with the height

measurement of the analyzed band. The use of band intensity ratio has been a solution for correcting intensity variation<sup>[34]</sup>. Band height correction via algorithm used by Datta et al.<sup>[47]</sup> may also have introduced a complex step to the analysis. The vinylidene band at 885 cm<sup>-1</sup> is characteristic only of NR and have also been tested in this study, which was not accomplished in the aforementioned investigation.

NR/SBR/BR contents determination is performed in the MIR region by transmission/controlled pyrolysis without extraction<sup>[14]</sup> between 450 and 400 °C. The pyrolyzed sample is analyzed as a liquid film. The bands are measured by intensity/height choosing appropriate baselines. SBR content is determined through the calibration curve involving the band intensity ratio ( $A_{699}/A_{1375}$ ) versus SBR concentration. BR content is determined through the calibration curve involving the band intensity ratio ( $A_{967}/A_{1375}$ ) versus BR concentration. In addition to IR analysis, TGA, differential scanning calorimetry (DSC) and gas chromatography (GC) coupled with pyrolysis mass spectrometry (Py-GC/MS) are also applied. Among them, the quantitative analysis using Py-GC/MS is considered the most precise of others techniques. Py-GC/MS provided more precise results, as the others techniques were affected by the elastomers interference. A considerable variation in content determination was observed, particularly with TGA and IR analyses. The analytical bands used (cm<sup>-1</sup>) are: 1375 (NR), 699 (SBR), and 967 (BR - trans C=C).

Although the research by Lee et al.<sup>[14]</sup> also showed adequate results for the determination of NR/SBR/BR, more than one instrumental technique was used. Although the Py-GC/MS have demonstrated greater precision, employing coupling techniques requires specialists in both areas, adding complexity to the methodology. The vinylidene band at 885 cm<sup>-1</sup>, characteristic of NR, also was not tested. Additionally, the BR band at 967 cm<sup>-1</sup> overlaps with the band of the same group in SBR. Moreover, the appropriate use of band intensity ratio could have reduced the IR methodology error. The lowest error found in the methodology was 5%.

Consequently, this scenario motivates the development of IR methodologies applying transmission and reflection (UATR) modes, with samples prepared by pyrolysis in a Bunsen burner and the selection of non-overlapping bands for the NR/SBR/BR content determination. Calibration curves are created with the median IR absorbance values from the analytical bands chosen for each elastomer versus its content in the blend. The errors involved are assessed, in accordance with a specific statistical treatment for IR spectroscopy<sup>[49]</sup>, as used in recent studies<sup>[29,33,34]</sup>. Acid-resistance tests<sup>[50]</sup> are able to differentiate saturated from unsaturated rubbers and are typically used in qualitative investigations, therefore were employed in this paper quantitatively to demonstrate the effectiveness of the developed IR methodology. This test is used as a quantitative indicator, as an original contribution. So, this investigation contributes to the state of the art in quantitative elastomer research by FT-IR techniques and simple laboratory tests.

## 2. Materials and Methods

### 2.1 Materials

NR/SBR/BR samples were prepared by Tenneco Automotive Brazil: 10NR/70SBR/20BR, 20NR/10SBR/70BR,

25NR/25SBR/50BR, 50NR/25SBR/25BR, and 70NR/20SBR/10BR. The numbers cited in the sample coding are associated with the weight content (%) of each elastomer in the ternary blend. These samples were used to construct the calibration curves for the FT-IR methodology. For the acid-resistance methodology, in addition to these samples, the one containing 25NR/50SBR/25BR was also used to evaluate the BR content.

The data regarding the rubber's formulation, including their additives, were encoded by the supplier. Therefore, it was not provided in this work, nor were the processing details sent due to internal company policies. However, the data relevant to the methodology development are only the contents of each elastomeric component, which have already been reported. Regarding the additives, as they are extracted with solvent, they do not interfere in the NR/SBR/BR determination.

## 2.2 IR Methodologies (transmission and reflection/UATR and acid-resistance) for NR/SBR/BR

The conditions for the IR analyses were: PERKINELMER IR spectrometer Frontier, 4000 to 400  $\text{cm}^{-1}$ , resolution 4  $\text{cm}^{-1}$ , gain 1, and 20 scans by transmission and reflection (UATR). In both methodologies, the samples were pyrolyzed in a Bunsen burner, after solvent extraction in acetone, and then analyzed as liquid films. The analytical bands used in both methodologies were selected at the following wavenumbers ( $\text{cm}^{-1}$ )<sup>[42]</sup>:  $A_{887}$  (vinylidene) for NR and  $A_{699}$  (aromatic C-H) for SBR. The  $A_{1375}$  band (methyl) for NR, used by Datta et al.<sup>[47]</sup> and Lee et al.<sup>[14]</sup>, was also tested. The BR band at 743  $\text{cm}^{-1}$  (cis C=C) was not used because of its low intensity, even though it would have been the typical choice due to its absence of overlapping with the SBR absorptions. The low intensity did not allow a proper height measurement, especially in sample with low BR content. The BR content was determined by subtraction using the obtained content of NR and SBR. The baseline ranges ( $\text{cm}^{-1}$ ) selected were: 930 to 852 for the 887 band; 712 to 680 for 699; and 1380 to 1350 for 1375. Analyses were performed in quintuplicate. The calibration curves were constructed with the median<sup>[49]</sup> values of absorbance versus the elastomer content. The accuracy estimation is in accordance with the nonparametric statistical method used for spectroscopic data<sup>[49]</sup>, successfully used for IR spectroscopic data in different studies<sup>[4,24,29,30,32,36,51,52]</sup>. The error of the developed methodology is estimated by the median of the relative errors<sup>[52]</sup>.

The following set of results were used for selecting the best analytical band or the best band intensity ratio: the highest linearity of the calibration curve ( $R^2$ ) and the lowest methodology error.

To verify the effectiveness of the FT-IR developed methodologies, three samples with similar content of NR/SBR/BR to those employed to construct the calibration curves were coded as samples  $A_1$ ,  $A_2$  e  $A_3$  and sent to the laboratory. The analyst was unaware of their nominal contents. To facilitate data evaluation, the tables with the results show the nominal contents of the test samples. The analysis conditions were the same as those used to construct the calibration curves.

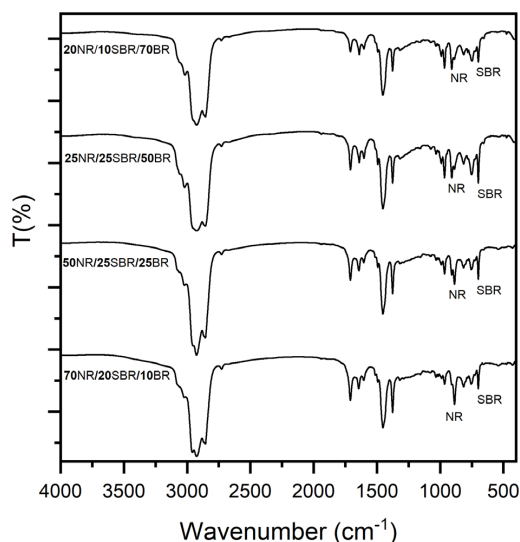
The acid-resistance test was also used for the IR data validation in this study. It was performed according to the methodology described in a previous investigation<sup>[50]</sup>. For the acid-resistance test<sup>[50]</sup>, which is an additional method for IR data validation, small sample fragments were extracted using acetone and subsequently dried in an oven. A mixture of concentrated  $\text{H}_2\text{SO}_4$  and concentrated  $\text{HNO}_3$  in a 1:1 volume ratio was introduced into a test tube immersed in water at 70 °C. Once the test tube mixture reached 70 °C, dried sample fragments were added. The time taken for the initiation of material degradation was then measured, observable when sample particles start to disperse. Since immediate degradation occurred at 70 °C, the test was repeated at 40 °C. Tests were conducted in triplicate, and the mean value was used to obtain a correlation between the degradation onset time and the BR content.

The acid-resistance test was conducted in triplicate. The mean time was used for evaluating the relation between the onset time of degradation and the BR content. The obtained acid-resistance data were also used to validate the BR contents in test samples, calculated by the IR methodology. To verify the effectiveness of the developed FT-IR or acid-resistance methodologies, the NR/SBR/BR samples coded " $A_1$ " (UATR and transmission FT-IR acid-resistance test) and " $A_3$ " (acid-resistance methodology) were analyzed by the same analysis conditions of samples used. In FT-IR methodologies, the median absorbance value of the analytical band was applied to the equation of the plotted calibration curve, corresponding.

## 3. Results and Discussions

### 3.1 Qualitative analysis (IR/UATR and IR/transmission) of the pyrolysed NR/SBR/BR blend

Figure 1 showed the spectra of IR/transmission/Bunsen burner pyrolysis of some NR/SBR/BR samples. The spectra were arranged according to the increase in the NR content to



**Figure 1.** IR/transmission/pyrolysis spectra of NR/SBR/BR samples.

demonstrate that as the content of this elastomer increases, the intensity of the band increases at 887 cm<sup>-1</sup> (indicated by the acronym NR).

Although the nominal SBR content does not increase in a regular way in the samples, it is also possible to observe that there is a variation in the intensity of the band at 699 cm<sup>-1</sup> (indicated by the acronym SBR) according to the content of this elastomer. Therefore, both bands at 887 cm<sup>-1</sup> and at 699 cm<sup>-1</sup> obey the Lambert-Beer law<sup>[42]</sup> and can be considered analytical, which means that they can be associated with the elastomer content and be considered suitable for the determination of the elastomers content in the blend.

The ternary blend NR/SBR/BR is considered a somewhat complex system<sup>[14]</sup>, as overlapping IR absorptions may occur, such as those of the C=C vinyl and trans groups of BR and SBR, which can be observed in the IR spectra of the pyrolyzed blends compared to the spectra of their elastomers (Figure 2). Bands of C=C vinyl and trans<sup>[42]</sup> groups (1000-900 cm<sup>-1</sup>) are present in SBR and BR. The band of the C=C cis group<sup>[42]</sup>, around 740 cm<sup>-1</sup>, which does not present any overlap, is of low intensity. Therefore, it was chosen, in this study, to calculate the NR and SBR contents and obtain the BR content by subtraction, as shown in the discussion of the quantitative analysis.

3.2 Quantitative IR/UATR/pyrolysis analysis of NR/SBR/BR

The IR/UATR results of the NR/SBR/BR blend pyrolysed in the Bunsen burner were plotted in a calibration curve for the A<sub>887</sub>/A<sub>698</sub> relative band (NR and SBR) versus the NR content. The band intensity ratio was used to reduce the effect of the variation in band thickness and height measurements. The methodology error was around 4%, lower than the reported in the literature (5%)<sup>[14]</sup> (Table 1). Then, the accuracy is considered satisfactory when comparing with the literature and under the conditions used. In addition, this

methodology error could be acceptable in the industry, from a technological perspective, where a specification range is routinely adopted for material acceptance. Although it is above the reference value for the equipment precision limit<sup>[49]</sup> (≤ 2%), this reference error is more commonly found in transmission analyses with thickness control. Even though the methodology showed some limitation, having only responded to four samples, a tendency in linearity was observed in Figure 3A (Equation 1), with 79% of the data explained by the developed methodology (R<sup>2</sup>).

y = 0.0085x + 0.1932 (1)

where y is the median absorbance value of the analytical band intensity or of the band ratio and x is the elastomer content corresponding for all calibration curves.

Another calibration curve with the SBR analytical band 699 cm<sup>-1</sup> versus the SBR content was plotted. See details in

Table 1. IR(A<sub>887</sub>/A<sub>698</sub>)/UATR/pyrolysis results for the determination of NR in NR/SBR/BR.

NR/SBR/BR (relative content)	A <sub>887</sub> /A <sub>698</sub>	Parameters
10NR/70SBR/20BR	0.155	$\hat{\mu} = 0.147$
	0.155	$R = -0.014$
	0.147	$\hat{\sigma} = 0.006$
	0.143	$\hat{\sigma}_{\hat{\mu}} \approx 0.003$
	0.141	$RD = 2.04\%$
20NR/10SBR/70BR	0.571	$\hat{\mu} = 0.513$
	0.526	$R = 0.112$
	0.513	$\hat{\sigma} = 0.048$
	0.472	$\hat{\sigma}_{\hat{\mu}} = 0.021$
	0.459	$RD = 4.09\%$
50NR/25SBR/25BR	0.564	$\hat{\mu} = 0.633$
	0.500	$R = 0.144$
	0.633	$\hat{\sigma} = 0.062$
	0.644	$\hat{\sigma}_{\hat{\mu}} = 0.028$
	0.644	$RD = 4.42\%$
70NR/20SBR/10BR	0.547	$\hat{\mu} = 0.750$
	0.750	$R = 0.218$
	0.765	$\hat{\sigma} = 0.094$
	0.621	$\hat{\sigma}_{\hat{\mu}} = 0.042$
	0.750	$RD = 5.60\%$

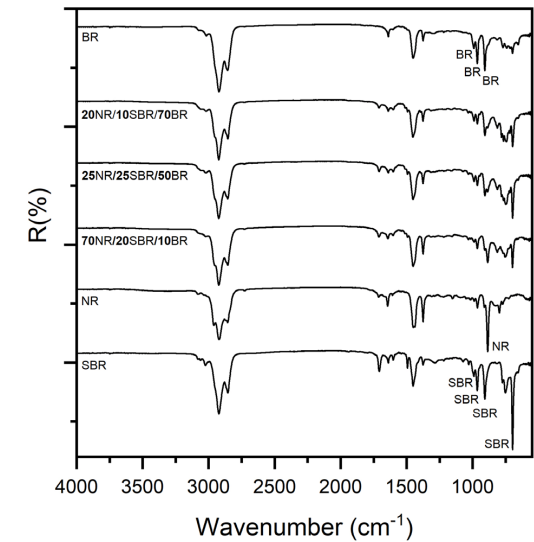
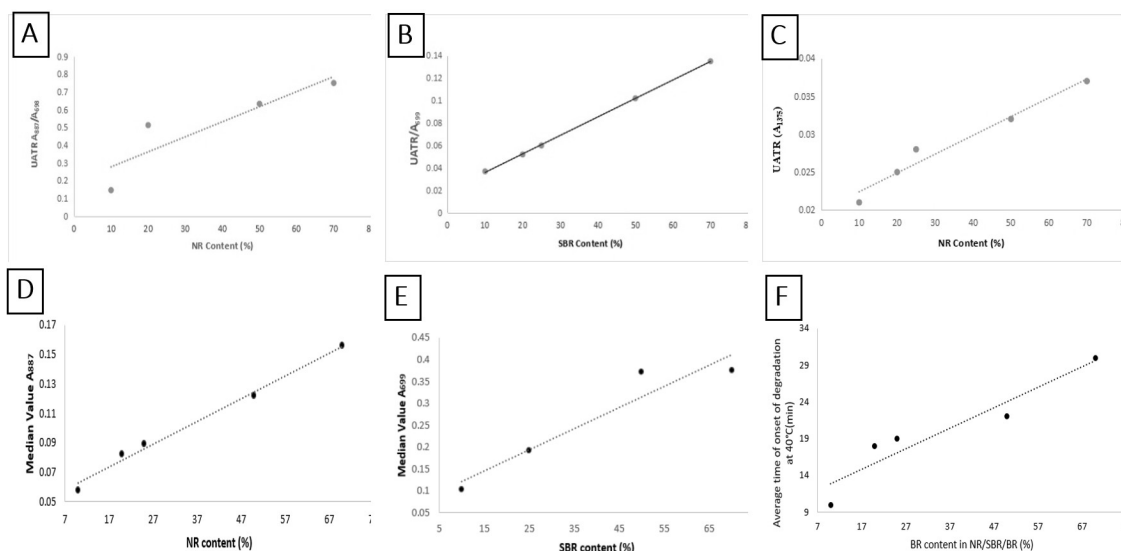


Figure 2. IR/UATR/pyrolyzed spectra of NR/SBR/BR and their elastomers.





**Figure 3.** Calibration Curves: (A)  $IR(A_{887}/A_{699})/UATR$  versus NR content; (B)  $IR(A_{699})/UATR$  versus SBR content; (C)  $IR(A_{1375})/UATR$  versus NR content; (D)  $IR(A_{887})/transmission$  versus NR content; (E)  $IR(A_{699})/transmission$  versus SBR content; and (F) Acid-resistance test - initial degradation time (min) at 40 °C versus BR content (%).

Table S1 of the Supplementary Material. The BR content was calculated by subtraction (Equation 2):

$$NR + SBR + BR = 100\% \quad (2)$$

This step of the study was developed differently from the methodologies found in the literature to lessen measurement errors, including the validation of the obtained BR content by acid resistance. Even though Datta et al.<sup>[47]</sup> used the 738  $\text{cm}^{-1}$  band, which does not present overlap, for the BR estimation, the band height measurement was corrected by an algorithm, which introduces a complex step in the analysis. The methodology error of Datta et al.<sup>[47]</sup> was not discussed. Lee et al.<sup>[14]</sup> used the band at 967  $\text{cm}^{-1}$  (trans C=C) for the BR determination, however this band overlaps with the absorption of the same functional group present in SBR. They reported that the lowest error for the IR methodology was 5%. The Py-GC/MS methodology, which involves coupling techniques, presented the highest precision. The band at 1375  $\text{cm}^{-1}$ , which is also present in other elastomers, and was investigated by Datta et al.<sup>[47]</sup> and Lee et al.<sup>[14]</sup> for NR, was also tested in this study (results in Table S2 of the Supplementary Material). For the SBR content determination, the height measurements for the band at 699  $\text{cm}^{-1}$  and the calculation of the methodology error were performed in the same way of those in Table 1. The median values of  $A_{699}$  versus the SBR content in NR/SBR/BR were plotted (Figure 3B, Equation 3). For the remainder of this study, the same type of calculation was adopted and, therefore, only the calibration curves are shown.

$$y = 0.0016x + 0.0196 \quad (3)$$

An adequate data correlation was observed ( $R^2 = 99\%$ ), which combined with an error of 1.67% (within the equipment precision limit<sup>[49]</sup>) indicates that the developed methodology can be used for the SBR determination in NR/SBR/BR.

To evaluate the NR band  $A_{1375}$ , used by Datta et al.<sup>[47]</sup> and Lee et al.<sup>[14]</sup>, a calibration curve was also plotted (Figure 3C, Equation 4). The adequate linearity of the calibration curve ( $R^2 = 96\%$ ) combined with an error of 3.57% indicated that the band  $A_{1375}$  can be used for the NR determination.

$$y = 0.0002x + 0.0199 \quad (4)$$

The comparison of the UATR calibration curves (Figure 3A and 3C) indicates that the curve plotted with the band  $A_{1375}$  incorporated a higher number of points and showed a superior linearity ( $R^2 = 96\%$ ). Consequently, it is more suitable for the NR determination in NR/SBR/BR using UATR/pyrolysis in a Bunsen burner. The methodology was evaluated by testing samples.

### 3.3 Quantitative IR/UATR/pyrolysis analysis on test samples

Table 2 shows the results of sample  $A_1$  for the determination of NR and SBR content using the band intensity ratio  $A_{887}/A_{699}$  and the absorption  $A_{699}$ . The calculated value for NR was relatively close to the nominal value. This is especially useful from a technological perspective, as previously discussed, for rubber companies that work with specification ranges. The calculated SBR content is very close to its nominal value because its analytical band at 699  $\text{cm}^{-1}$  is strong, therefore it is more suitable for the height measuring<sup>[42]</sup>, even for low content of SBR, indicating suitable precision. The calculated BR content (by Equation 2) was 30.5% (nominal, 25%), which according to technological aspects is considered satisfactory.

Table 3 presents the results obtained for the NR and the SBR content determination in sample  $A_2$  using the  $A_{1375}$  band for NR and the  $A_{699}$  band for SBR. The calculated NR content is close to the nominal value.

**Table 2.** Results of sample A<sub>1</sub> for the IR/UATR (A<sub>887</sub>/A<sub>699</sub>) determination of the NR content.

Sample	A <sub>887</sub> /A <sub>699</sub>	$\frac{A_{887}/A_{699}}{\text{(median)}}$	NR calculated content (%) Equation 1	NR nominal content (%)
A <sub>1</sub>	0.678	0.564	43.62	50
	0.557			
	0.564			
	0.524			
	0.638			
Sample	A <sub>699</sub> (SBR)	$\frac{A_{699}}{\text{(median)}}$	SBR calculated content (%) Equation 3	SBR nominal content (%)
A <sub>1</sub>	0.059	0.061	25.88	25
	0.061			
	0.062			
	0.063			
	0.058			

**Table 3.** Results of sample A<sub>2</sub> for the IR/UATR (A<sub>1375</sub>/) determination of the NR content.

Sample	A <sub>1375</sub> (NR)	$\frac{A_{1375}}{\text{(median)}}$	NR calculated content (%) Equation 4	NR nominal content (%)
A <sub>2</sub>	0.025	0.025	25.5	20
	0.026			
	0.025			
	0.025			
	0.025			
Sample	A <sub>699</sub> (SBR)	$\frac{A_{699}}{\text{(median)}}$	SBR calculated content (%) Equation 3	SBR nominal content (%)
A <sub>2</sub>	0.035	0.036	10.25	10
	0.035			
	0.036			
	0.036			
	0.036			

The calculated content for the BR rubber was 64.25%, close to the nominal value of 70%, according to a technological point of view. In addition, the results suggest that the bands A<sub>887</sub>/A<sub>699</sub> and A<sub>1375</sub> can be used for the determination of the NR content in NR/SBR/BR. In this study, the same NR/SBR/BR blend was also investigated by transmission/pyrolysis. The difference in the optical path in transmission (optical path constant) and reflection (optical path variable) modes may influence the precision of results.

3.4 Quantitative IR/transmission/pyrolysis analysis of NR/SBR/BR

For the transmission analysis, it was decided to evaluate only the band at 887 cm<sup>-1</sup> for NR, without overlap, since similar results were observed for the band at 1375 cm<sup>-1</sup> in the reflection methodology (UATR). An adequate linearity was observed (calibration curves - Figure 3D – NR, Equation 5, R<sup>2</sup> = 99% and Figure 3E – SBR, Equation 6, R<sup>2</sup> = 91%). Refer to Tables S3 and S4 in the Supplementary Material for detailed results.

y = 0.0016x + 0.0472 (5)

y = 0.0049x + 0.0721 (6)

The error for the methodology involving bands A<sub>887</sub> and A<sub>699</sub> was approximately 6%. It is similar to the one reported in the literature<sup>[14]</sup>, 5%, which was obtained using controlled pyrolysis that involves the use of additional accessories, thereby increasing the time and cost of analysis. Thus, considering the conditions of analysis used in this study, i.e., sample pyrolyzed in a Bunsen burner without control of temperature and the comparative error (5% - controlled pyrolysis<sup>[14]</sup> and 6% - pyrolysis in a Bunsen burner), these results are assessed as satisfactory.

3.5 Effectiveness of the IR/transmission/Bunsen Burner pyrolysis methodology

Another test sample (A<sub>3</sub>) was analyzed under the same conditions as the calibration curves. Table 4 presents the results regarding the determination of the NR and SBR content using the bands A<sub>887</sub> and A<sub>699</sub>. The calculated NR and SBR contents were close to the nominal value, with the SBR determination showing higher precision. The calculated BR content was 28.61%, close to the nominal value of 25%.

### 3.6 Acid-resistance test and validation of the quantitative IR methodology for BR

The acid-resistance test<sup>[50]</sup> was conducted to verify the quantitative relation between the increase in BR content and the degradation onset time of the NR/SBR/BR blend, as this evaluation might help validating the IR methodology in which the BR content was obtained by subtraction. According to Dutra and Diniz<sup>[50]</sup>, the unsaturated rubbers NR, SBR, and BR degrade within  $\leq 1$  minute at 70 °C. Therefore, the NR/SBR/BR blend is expected to have a quick degradation at 70 °C, making it impossible to establish any relation between the BR content and the degradation time. For this reason, the test was conducted at 40 °C (Table 5). At 40 °C, the longer time required to start the degradation of each rubber in the blend permits a proper individual visualization. Moreover, according to the literature, the elastomers combined in the blend have different degradation onset time at 40 °C. NR rubber degrades in less than 3 minutes, while BR and SBR degrade between 10 and 30 minutes. Results were used to plot a calibration curve that establishes a relation between the BR content in the ternary rubber and the average time required for the onset of degradation at 40 °C (Figure 3F). It was observed a correlation (Equation 7) with an adequate linearity ( $R=0.946$  and  $R^2=90\%$ ) between the data obtained through the acid-resistance tests and the BR content in the sample.

$$y = 0.2792x + 10.029 \quad (7)$$

where, y is the degradation onset average time at 40 °C and x is the BR content in NR/SBR/BR.

The acid resistance analysis of BR at 40 °C showed a longer degradation onset time compared to NR<sup>[50]</sup>. Figure 3F

demonstrated that the higher the BR content in the blend, the longer the material degradation onset time. To further validate the developed IR methodology and emphasize its effectiveness, acid-resistance tests were conducted at 40 °C in two blend test samples containing 25% of BR (25NR/50SBR/25BR – A<sub>3</sub> and 50NR/25SBR/25BR – A<sub>1</sub>). The latter was also analyzed as a test sample for the IR methodologies (transmission and reflection). Both samples exhibited the same degradation onset time: approximately 19 minutes at 40 °C. Therefore, these acid-resistance results can be related to those obtained by the IR methodologies, where the BR content was calculated by subtraction and presented values relatively close to the nominal 25% (28.61% by transmission/pyrolysis, 30.50% by UATR/pyrolysis and 32% by acid-resistance). Although, as expected, the results of the acid-resistance test are less accurate than those of instrumental techniques, the results are considered satisfactory for sectors that work with a material specification range, as already explained.

### 3.7 NR/SBR/BR FT-IR data validation–MIR/reflection (UATR) and MIR/transmission

In this study, two spectra obtaining modes - transmission and reflection - were used. For data validation, a sample (A<sub>1</sub>) containing the same nominal content (50NR/25SBR/25BR) was analyzed using both methodologies, with NR band at 887 cm<sup>-1</sup> and SBR at 699 cm<sup>-1</sup> (Table 6).

In both methodologies, the calculated results for the SBR content were the closest to the nominal value, probably due to the intense absorption of the band at 699 cm<sup>-1</sup>, which facilitates the measurement of the analytical band height. The values obtained in both analyses were close to each other and to the nominal content, especially in the

**Table 4.** Results of sample A<sub>3</sub> for the IR(A<sub>887</sub>)/transmission/ determination of NR content.

Sample	A <sub>887</sub> (NR)	A <sub>887</sub> (median)	NR calculated content (%)	NR nominal content
			Equation 5	(%)
A <sub>3</sub>	0.097	0.120	45.5	50
	0.132			
	0.098			
	0.120			
	0.153			
Sample	A <sub>699</sub> (SBR)	A <sub>699</sub> (median)	SBR calculated content (%) Equation 6	SBR nominal content (%)
A <sub>3</sub>	0.153	0.199	25.89	25
	0.199			
	0.144			
	0.202			
	0.240			

**Table 5.** Results of acid-resistance (40 °C) for the NR/SBR/BR blend.

Sample	Temperature (°C)	Time (min)	Average (min)
70NR/20SBR/10BR	40	11/9/10	10
10NR/70SBR/20BR		18/18/19	18
25NR/50SBR/25BR		15/19/21	18
50NR/25SBR/25BR		17/18/21	19
25NR/25SBR/50BR		20/22/23	22
20NR/10SBR/70BR		28/31/32	30

**Table 6.** UATR and transmission methodologies for the NR/SBR/BR determination.

Spectra obtaining modes	NR content (%)	SBR content (%)	BR content (%)	NR/SBR/BR nominal content (%)	Methodology error (%) (transmission)	Methodology error (%) (UATR reflection)
Transmission	45.50	25.89	28.61	50/25/25	6	4
UATR reflection	43.62	25.88	30.50			

transmission methodology. Nevertheless, the reflection/UATR methodology showed a better precision in terms of data repeatability, with a lower error of 4%, compared to the transmission with 6% error, and also according to the findings of Lee et al.<sup>[14]</sup>, that reported 5% error.

The comparison of the methodology error by Lee et al.<sup>[14]</sup> (5%), with the lowest value found by IR/UATR in this study (4%), although similar, shows that the objective of finding a most appropriate error was achieved, as it was not necessary to use an accessory for controlled pyrolysis, a technique that had been used in the literature<sup>[14]</sup>. The methodology developed in this investigation demonstrates that a simple Bunsen burner, found in all laboratories, is enough to achieve good results, reducing costs and also providing a slight improvement in precision with the use of relative band. This study also presents an alternative to using the analytical band with overlap to estimate the BR content, which is the calculation by subtraction in relation to the NR and SBR content. The IR results were validated by acid resistance, the use of which as quantitative data is presented in this study as a novel approach.

#### 4. Conclusions

This investigation goal was to develop a simple and precise IR methodology for the quantitative IR analysis of the NR/SBR/BR rubber blend, based on different spectra obtaining modes (transmission and UATR reflection), and also to introduce the undemanding acid-resistance test for routine quantitative analysis. The samples were prepared for IR analysis using the same technique: pyrolysis in the Bunsen burner. Non-overlapping bands were selected to calculate the content of each elastomer, achieving better results. Both methodologies can be rapidly applied for elastomer content determination, with a slight advantage in terms of precision for the reflection/UATR methodology compared to literature data. The methodologies were validated through test samples and quantitative acid-resistance tests. The error of the developed methodology could be acceptable in the industry, from a technological perspective, where a specification range is routinely adopted for material acceptance. Hence, the objective of this study was achieved, as an alternative methodology to that found in the literature was presented, with more accessible conditions, lower cost, and with more precise results. In addition, it was presented for the first time the validation of IR data with the use of quantitative acid-resistance analysis of a ternary rubber. These findings contribute to the state of the art in the determination of elastomers content.

#### 5. Author's Contribution

- **Conceptualization** – Alexandra Helena de Barros; Rachel Farias Magalhães; Rita de Cássia Lazzarini Dutra.
- **Data curation** – NA.
- **Formal analysis** – NA.
- **Funding acquisition** - Rita de Cássia Lazzarini Dutra.
- **Investigation** - Milton Faria Diniz; Alexandra Helena de Barros; Rachel Farias Magalhães.
- **Methodology** – Alexandra Helena de Barros; Rachel Farias Magalhães; Milton Faria Diniz; Rita de Cássia Lazzarini Dutra.
- **Project administration** – Rita de Cássia Lazzarini Dutra.
- **Resources** –Lídia Mattos Silva Murakami.
- **Software** – NA.
- **Supervision** – Natália Beck Sanches; Jorge Carlos Narciso Dutra; Rita de Cássia Lazzarini Dutra.
- **Validation** – Alexandra Helena de Barros; Rachel Farias Magalhães; Taynara Alves de Carvalho; Rita de Cássia Lazzarini Dutra.
- **Visualization** – Natália Beck Sanches; Rita de Cássia Lazzarini Dutra.
- **Writing – original draft** –Alexandra Helena de Barros; Rachel Farias Magalhães; Rita de Cássia Lazzarini Dutra.
- **Writing – review & editing** – Alexandra Helena de Barros; Rachel Farias Magalhães; Taynara Alves de Carvalho; Rita de Cássia Lazzarini Dutra.

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## **Supplementary Material**

Supplementary material accompanies this paper.

Table S1. FT-IR/UATR(A699) data/Bunsen Burner pyrolysis for SBR determination in NR/SBR/BR.

Table S2. FT-IR (A1375) data/UATR/Bunsen Burner pyrolysis for NR determination in NR/SBR/BR.

Table S3. FT-IR data/transmission/Bunsen Burner pyrolysis/film with cover slip aid for NR determination in NR/SBR/BR - band A887.

Table S4. FT-IR data/transmission/Bunsen Burner pyrolysis/film with cover slip aid for SBR determination in NR/SBR/BR - band A699.

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