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Study of the Consumption of the Curing Agent on the Liner Surface by Infrared Spectroscopy in Reflection Mode

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ABSTRACT

Monitoring the reaction between the isocyanate group (NCO) of the curing agent 2,4-toluene diisocyanate and the hydroxyl group (OH) of hydroxyl-terminated polybutadiene is important for the rocket engine loading process, since the reduction of NCO content on the adhesive surface (liner) directly affects adhesion to the propellant. Traditionally, the tack point, which represents the optimal time window for rocket motor loading, is determined based on the operator's tactile perception. Viscosimetry is employed to monitor adhesive curing and subsequently assess tack; however, as this method evaluates the reaction within the bulk material, adhesion control remains predominantly empirical when chemical reference parameters are preferred. This study proposes a methodology based on infrared spectroscopy (IR) in universal attenuated total reflection mode, evaluating four stoichiometric ratios R(NCO/OH): R = 1.00 and, with excess NCO, R = 1.05, 1.10, and 1.15. The ratio R = 1.05 showed the best performance, with a methodological error of 4%. The main contribution of this work is a fast and precise IR surface analysis that enhances process control during solid rocket motor loading.

Keywords: Adhesives; Curing; Infrared spectroscopy; Isocyanates; Surfaces.

INTRODUCTION

Solid propellant rocket motors comprise three main systems: ignition, nozzle, and case. Among these components, the case houses the solid fuel, also referred to as composite solid propellant or simply solid propellant (Mason and Roland 2019).

Solid propellant is a heterogeneous mixture composed of a polymeric binder, metallic fuels, inorganic oxidizers, curing agents, and other additives. Each component has specific functions and, together, during combustion, generates large amounts of energy as heat and gases, producing the thrust required to propel the rocket (Naseem *et al.* 2021; Yadav *et al.* 2021).

Regarding the casting process, solid propellants are classified into two major categories: case-bonded and cartridge-loaded (Ghosh *et al.* 2021). In the case-bonded type, the propellant remains adhered to the inner wall of the combustion chamber, usually

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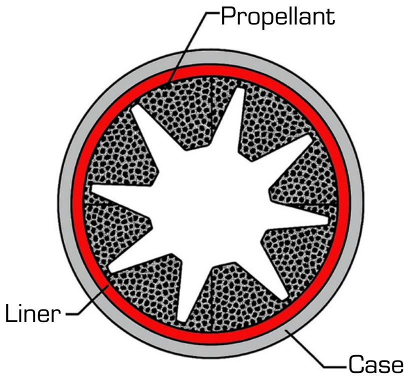
by an adhesive. In the cartridge-loaded type, the propellant is molded externally and, once finalized, with or without an inhibitor layer, is inserted into the case (Ashish *et al.* 2019; Ghosh *et al.* 2021; Mason and Roland 2019).

For rocket motors whose propellant is case-bonded, the internal structure includes two additional components: an ablative thermal insulator to protect the case from the severe temperature, heat flux, and mass flow generated during propellant combustion, and an adhesive to bond the propellant to the thermal insulator (Amado *et al.* 2020; 2022; Mosa *et al.* 2022). The adhesive layer that coats the motor interior and whose primary function is to keep the solid propellant stable inside the case after curing is commonly called the liner. It acts as an adhesive interface between the thermal insulator and the propellant, or between the case and the propellant, depending on the design (Amado *et al.* 2022; Mason and Roland 2019; Naseem *et al.* 2021).

The case-bonded casting process requires special attention to the adhesive interphase between the liner and the solid propellant to avoid debonding that may compromise propellant structural integrity and performance. Depending on the type and location of the adhesive failure, new burning areas may form, leading to uncontrolled propellant combustion (Amado *et al.* 2022; Nassen *et al.* 2021).

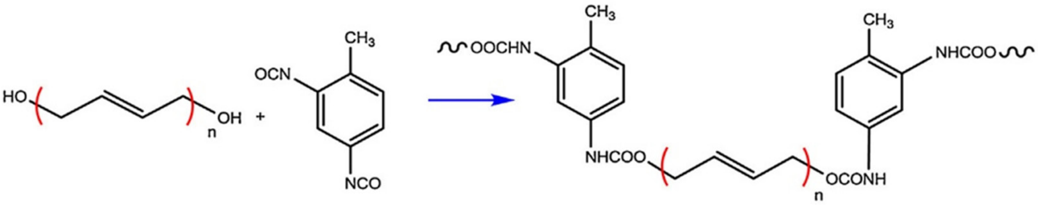
According to the American Society for Testing and Materials (ASTM) D 907-2005, an adhesive is a compound capable of keeping different materials joined by surface contact. In the aerospace sector, liner (Fig. 1) is the term used for a polyurethane-based adhesive (PU) applied to bond the propellant to the interior of the rocket motor case (Fig. 1) (Pinto and Dutra 2023).



Source: Adapted from Pinto and Dutra (2023).

Figure 1. Schematic of a rocket motor indicating the liner.

The liner is the product of the reaction between a resin and a curing agent, which promotes bonding among the polymer chains in the matrix. In this study, hydroxyl-terminated polybutadiene (HTPB) is the polymeric matrix and 2,4-toluene diisocyanate (TDI) is the curing agent, as shown in Fig. 2 (Toosi *et al.* 2015).



Source: Adapted from Toosi *et al.* (2015).

Figure 2. Curing reaction between HTPB and TDI.

Studies on liner-propellant adhesion date back to the 1960s, when Bills *et al.* (1966) and Gustavson *et al.* (1966) emphasized the importance of understanding the adhesion mechanisms to prevent failures during casting, curing, storage, and launch. Since then, a widely accepted theory has been chain interdiffusion between the polymer matrices of the propellant and the liner (Gustavson *et al.* 1966).

According to the interdiffusion principle, molecules display high mobility across interfaces, especially while the adhesive and propellant are not fully cured. Given the liner's relevance in the casting process, monitoring its curing is essential to determine the appropriate moment to insert the propellant into the case, promoting the formation of a three-dimensional interphase with properties distinct from the original phases that maximizes adhesion to the liner. Therefore, establishing the curing curve, that is, the kinetic behavior of liner curing, is essential (Pinto and Dutra 2023).

In this context, monitoring the reaction between the functional isocyanate group (NCO) of TDI and the hydroxyl group (OH) of HTPB is important because the reduction in NCO availability during curing directly affects adhesion between the liner and the propellant, whose polymer matrix is the same resin as the adhesive (HTPB). Thus, the propellant provides OH groups for urethane bond formation, which promotes the interphase and the resulting adhesion process (Pinto and Dutra 2023).

Currently, viscosimetry is used to monitor liner curing. However, this technique does not adequately reflect the decrease in NCO groups at the material surface, which governs adhesion, so the determination of the ideal moment to add the propellant to the rocket motor remains largely empirical and dependent on operator sensitivity (Pinto 2007).

The contents of OH and NCO can be determined by Fourier transform infrared spectroscopy (FT-IR) (Takahashi *et al.* 1996) using analytical bands associated with the groups of interest, around 3610 cm^{-1} (OH) and 2270 cm^{-1} (NCO), respectively (Smith 1979). In this approach, methodological errors remain within the equipment precision limit, about 2%, in accordance with the statistical treatment recommended for FT-IR analyses (Hórak and Vítek 1978). In general, spectra are acquired in the mid-infrared (MIR) region using transmission mode.

More recent studies (Brito and Fonseca 2021; Siqueira *et al.* 2008) have investigated the determination and consumption of NCO in polyurethane adhesives using reflection techniques for FT-IR spectra acquisition. The determination of isocyanates in the near-infrared (NIR) region was also evaluated by Siqueira *et al.* (2008), with methodological error around 2%, a reference value under ideal thickness control in transmission, consistent with Hórak and Vítek (1978).

In a recent study (Kovács *et al.* 2024) on the effect of environmental conditions on the curing of a polyurethane adhesive, FT-IR analysis in both transmission and reflection modes was used to evaluate spectrometric changes and temporal variation of band intensities. The work highlights the importance of adhesives across industries, market growth, and the critical role of time in adhesive bonding. The main objective was to study the crosslinking reaction, its rate, and how it varies under different environments. The results suggest that the crosslinking rate changes significantly with environmental conditions (room temperature [RT], and RT with 30% and 100% humidity), indicating that such factors should not be neglected in bonding. Consequently, the surrounding environment significantly influences adhesive application.

Although the study by Kovács *et al.* (2024) presents absorbance-versus-time curves, it does not explicitly indicate the evaluation of measurement errors or the quantification of reacted NCO over time, as performed in this work. This gap opens room for the contribution proposed here to the state of the art on polyurethane adhesive curing, especially at the surface.

Beyond Kovács *et al.* (2024), the state of the art includes the control of unreacted isocyanate in food packaging (Osipova 2024), a topic directly related to health risks. Aromatic molecules containing unreacted NCO can migrate through packaging layers and, upon contact with water in food, react to form carcinogenic primary aromatic amines. This need for control reinforces the development of fast and precise methodologies with clearly defined NCO quantification limits.

FT-IR analysis, due to its ability to characterize, identify, and quantify functional groups, depending on the acquisition mode and selected analytical band, is widely employed for this purpose in different materials, regardless of application. Therefore, it can be used to quantify NCO in both packaging materials and aerospace systems.

In the study by Osipova (2024), FT-IR spectra were collected in the MIR region in transmission mode using a horizontally mounted cell with a 100 μm optical path length. The analytical band of isocyanate was observed at 2268 cm^{-1}, assigned to the NCO stretching mode, the same used in the present work. Band heights were measured for each sample and, according to the Beer-Lambert law, were proportional to isocyanate concentration, enabling the construction of a calibration curve of absorbance versus NCO content, with $R^2 = 0.998$, meaning about 99% of the variance was explained by the methodology.

Although Osipova (2024) reports relevant results, including an adequate sample size ($n = 8$) and definitions of detection and quantification limits, understood as the smallest detectable amount and the smallest amount measurable with precision and accuracy, respectively, the statistical treatment used to calculate deviations is not described. The methodological error is also not reported and should be estimated using a spectroscopy-specific approach such as that of Hórak and Vítek (1978).

The literature also includes a review of modification methods, joint types, and self-healing strategies for adhesives in the aerospace industry (Hu *et al.* 2024). The study synthesizes the state of research on adhesive technology, addressing methods to improve bonding performance and process influences. The authors distinguish between extrinsic self-healing (microcapsules and nanoparticles) and intrinsic self-healing (reversible chemical bonds). Both approaches can promote self-healing of cracks in the adhesive layer but are sensitive to environmental conditions. From this perspective, the review emphasizes the need for ongoing, innovative research into modified adhesives. The self-healing mechanism reduces frequent joint replacement in the presence of fractures in the adhesive layer, and the synergistic development of mechanical properties and self-healing mechanisms offers broad application prospects in aerospace.

The detailed review (Hu *et al.* 2024) does not focus on curing monitoring using instrumental techniques, which highlights a gap and, therefore, an opportunity for the present study to contribute, including the application of FT-IR, to the state of the art in aerospace adhesives.

Given that qualitative and quantitative studies of adhesives and formulations for flexible thermal protection in rocket motors (Pinto *et al.* 2018) have been successfully conducted using FT-IR, including curing assessments (Janzen *et al.* 2021), the same technique is employed here. Specifically, surface analysis by universal attenuated total reflection (UATR) in the MIR region is evaluated to monitor liner curing. This approach contributes to the development of new adhesion procedures for solid propellants in rocket motors by providing FT-IR methodologies as alternatives to viscosimetry for appropriate tack point determination.

METHODOLOGY

Material characteristics and curing process

Upon receipt of the labeled reagents HTPB and TDI, they were weighed on an analytical scale according to the stoichiometric ratio between the two components, defined as R(NCO/OH). In this study, R was evaluated at four levels: 1.00, 1.05, 1.10, and 1.15. Values of R greater than 1 indicate an excess of TDI, the component that supplies the NCO groups monitored during curing.

Preparation of the material to be analyzed/FT-IR analysis conditions

After weighing the reagents, they were mixed in a beaker with a spatula together with the reinforcing filler (carbon black), added to adjust the mechanical properties of the resulting liner system. The mixture was then poured into test holders in a number equal to the planned spectral measurements, totaling 14, distributed over the liner curing time. The specimen consisted of a silicone rubber holder with a central hole containing an aliquot of the liner to be analyzed.

Once the mixture was distributed, one specimen was selected and taken directly to a PerkinElmer Frontier FT-IR spectrometer equipped with a UATR accessory for the initial analysis of the reaction mixture. Before the first acquisition, a background spectrum of laboratory air was recorded for automatic subtraction by the software. The UATR diamond crystal was then cleaned to avoid contamination, and, due to the adhesive nature of the system, the specimen was positioned to ensure complete contact of the liner with the crystal without applying torque, since the sample is non-solid. The surface spectrum of the initial sample was acquired in the MIR region (4000 to 400 cm^{-1}), monitoring the NCO band of interest at 2270 cm^{-1}.

The remaining holders containing the liners were immediately placed in an oven at 50 °C and removed one by one at predefined times for spectral acquisition. The times, in hours, were as follows: initial, 0.25, 0.5, 0.75, 1, 2, 3, 4, 5, 6, 7, 8, 24, and 48, with shorter intervals in the early stages due to the higher curing rate, and longer intervals later due to the decrease in reaction rate over time.

After all spectra were acquired, they were analyzed using the FT-IR software to determine the height of the analytical NCO band, measured with a baseline from 2422 to 2100 cm^{-1}. For each R-value defined in the study, three measurements were performed at each time point on different days.

The variation in NCO band height and the percentage of reacted NCO groups as a function of time were evaluated using curves, according to Eq. 1 (Dutra 1984; Janzen *et al.* 2021):

$$\text{Percentage of reacted NCO groups} = \left[{(A_0 - A_t)}\big/{A_0} \right] \times 100 \tag{1}$$

The methodology adopted (Eqs. 2-4) for calculating the errors of the developed methods was applied in accordance with the nonparametric treatment (Hórak and Vítek 1978), specific to IR, previously employed in earlier and recent studies (Barros *et al.* 2023; 2025; Carvalho *et al.* 2025; Janzen *et al.* 2021).

$$\hat{\sigma} = K_R \cdot R \tag{2}$$

$$\hat{\sigma}_{\hat{\mu}} = \frac{\hat{\sigma}}{\sqrt{n}} \tag{3}$$

$$RD\ (\%) = \left(\frac{\hat{\sigma}_{\hat{\mu}}}{\mu}\right) \cdot 100 \tag{4}$$

where $\hat{\sigma}$ is the standard deviation, K_R is the coefficient for three measurements ($K_R = 0.591$), used for reaction studies (Janzen *et al.* 2021), R is the range, that is, the difference between the largest and smallest absorbance values, $\hat{\sigma}_{\hat{\mu}}$ is the mean standard deviation, n is the number of measurements, and RD is the error (or relative deviation) expressed as a percentage. The parameter μ is the median absorbance value, since Hórak and Vítek (1978) emphasize that, when the number of measurements is small, the median (μ) should be used instead of the arithmetic mean. This yields more consistent results with smaller errors.

According to Dutra *et al.* (1996), the method error, defined as the median of the relative errors, is the most appropriate value (in percentage) to indicate the precision of the developed methodology, a procedure adopted in previous works (Barros *et al.* 2023; 2025; Carvalho *et al.* 2025; Janzen *et al.* 2021).

RESULTS AND DISCUSSION

Methodology characteristics

The oven temperature holding the reaction mixture was maintained at 50 °C to control the ongoing reaction and simulate operational conditions. Different percentages of NCO excess were selected to assess whether the excess would be uniformly distributed within the liner mixture. Accordingly, the amount of free isocyanate at the material surface was varied to favor adhesion between the liner and the propellant. The proposed percentages also enabled verification of whether the decay of the infrared analytical band associated with NCO consumption in the 2260-2270 cm^{-1} range followed the same trend across the four proportions. An additional objective was to determine the time to reach the tack point for each sample using viscosimetry as a reference. Tack corresponds to the initial contact force of an adhesive with the bonding surface under minimal time and pressure.

Accordingly, the aim was to identify the highest R-value at which the liner exhibits adhesive properties with the propellant compatible with the rocket motor casting parameters, while meeting the process time requirements in terms of achieving the tack point.

Quantitative FT-IR/UATR analysis of the liner curing process

Regarding the calculations based on absorbance values, this study adopted the criterion of presenting Table 1 containing the complete dataset and detailed methodology for the R(NCO/OH) ratio of 1.05, which yielded the most suitable results. Due to the large volume of data, the results for the other R(NCO/OH) ratios were summarized and grouped in Table 2. These include values related to the quantification limit (reaction time corresponding to the lowest measurable absorbance or the highest percentage of reacted NCO) and the error associated with each method, given that the calculation mechanism is the same. NCO consumption was evaluated by changes in the median height of the 2270 cm^{-1} band (Fig. 3). The percentage of reacted NCO over time for R(NCO/OH) = 1.05 is shown in Fig. 4, with contents calculated according to Eq. 1.

Table 1. FT-IR/UATR data (A_{2270}) for the liner curing reaction at R(NCO/OH) = 1.05.

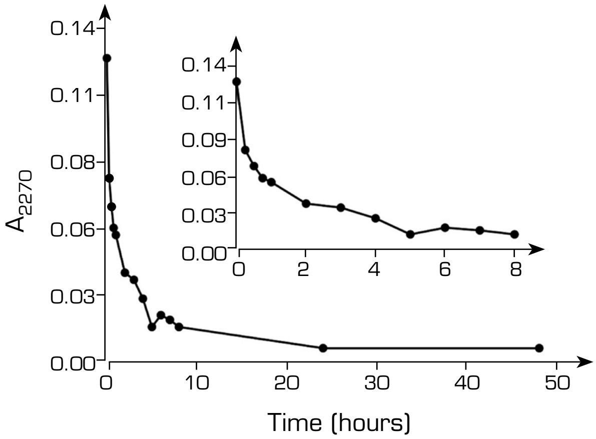
Time (hours)	A_{2270}	A_{2270} (median)	Mean standard deviation	Relative deviation* (%)	Reacted NCO groups*,** (%)
0.00	0.112	0.129	0.009	7	0
	0.129				
	0.140				
0.25	0.078	0.078	0.001	1	40
	0.074				
	0.078				
0.50	0.065	0.066	0.001	2	49
	0.066				
	0.066				
0.75	0.057	0.057	0.001	2	56
	0.059				
	0.057				
1.00	0.052	0.054	0.001	2	58
	0.055				
	0.054				
2.00	0.038	0.038	0.002	5	71
	0.043				
	0.037				
3.00	0.037	0.035	0.003	9	73
	0.035				
	0.027				
4.00	0.029	0.027	0.001	4	79
	0.027				
	0.027				
5.00	0.018	0.015	0.002	13	88
	0.015				
	0.011				
6.00	0.020	0.020	0.002	10	84
	0.022				
	0.016				
7.00	0.021	0.018	0.001	6	86
	0.018				
	0.018				
8.00	0.015	0.015	0.002	13	88
	0.019				
	0.013				
24.00	0.006	0.006	0.001	17	95
	0.007				
	0.004				
48.00	0.006	0.006	0.001	17	95
	0.006				
	0.005				

Source: Elaborated by the authors. *Values considered as integers. **Calculated according to Eq. 1. Methodological error: 4%.

Table 2. Comparison of FT-IR/UATR data (A_{2270}) for liner curing at different R(NCO/OH) values, including methodological errors and the quantification limit for reacted NCO.

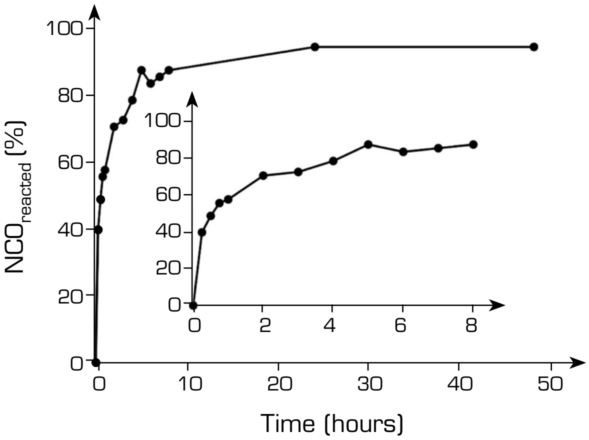
R(NCO/OH)	Quantification limit (hours)	% reacted NCO (approximately 8 h)	Methodological error (%)
1.00	6	90	5
1.05	5	88	4
1.10	4	79	7
1.15	5	79	3

Source: Elaborated by the authors.



Source: Elaborated by the authors.

Figure 3. Variation of the median value of the analytical band A_{2270} as a function of time for R(NCO/OH) = 1.05.



Source: Elaborated by the authors.

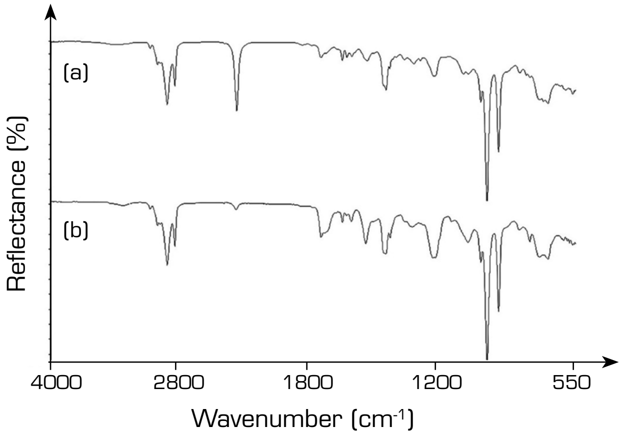
Figure 4. Percentage of reacted NCO groups as a function of time for R(NCO/OH) = 1.05.

An assessment of the Table 1 data indicates the presence of a quantification limit: after 5 h of reaction, accurate quantification of NCO content is no longer feasible due to the low intensity of the analytical A_{2270} band resulting from NCO consumption. Considering the nine relative deviation values at time points where measurements were possible, the methodological error was 4%, which is satisfactory under the employed conditions and close to the lower error bound reported by Janzen *et al.* (2021), 3%, for FT-IR studies of another aerospace adhesive using a similar calculation methodology.

Figure 3 confirms the decrease in the median absorbance values of the A_{2270} band, with minor variations likely associated with the quantification limit around 5 h of reaction. Regarding the percentage of reacted NCO groups (Fig. 4), FT-IR results indicate that at 5 h approximately 80% (median value in Table 2: 83.5%) of NCO groups had been consumed, a conversion level consistent with that measurable by FT-IR for another adhesive system (Janzen *et al.* 2021). The methodological error was also around 5% (median value in Table 2), consistent with Janzen *et al.* (2021), satisfactory for the employed conditions (surface analysis in triplicate), and within the reported range (3-7%).

Association between qualitative FT-IR spectral changes during liner curing and quantitative data

Figure 5 presents the FT-IR/UATR spectra at the initial time and after 24 h from the start of the experiment for R(NCO/OH) = 1.05.



Source: Elaborated by the authors.

Figure 5. FT-IR/UATR spectra obtained for the liner + TDI reaction mixture, for R(NCO/OH) = 1.05. A) Initial time; B) After 24 hat 50 °C in an oven.

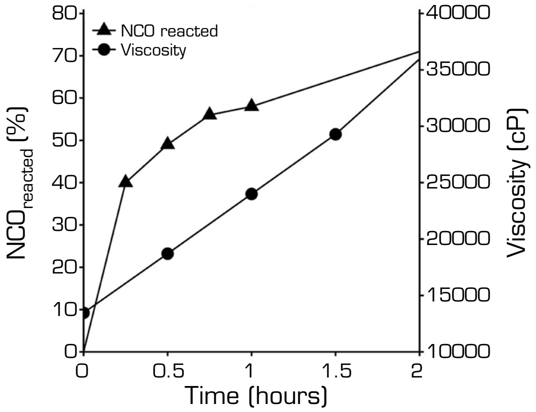
Analysis of the absorbance measurements and spectra indicates significant spectrometric changes consistent with the curing reaction. A decrease in the ~ 2270 cm^{-1} band was observed, associated with NCO consumption (the presence of a low-intensity band in the UATR spectrum indicates residual unreacted NCO at the surface); the appearance of bands at ~ 1530 cm^{-1} and ~ 3320 cm^{-1}, assigned to NH (amine); an increase in intensity at ~ 1730 cm^{-1}, assigned to carbonyl (C=O); and the emergence of a band at ~ 1220 cm^{-1} (C-O), both associated with ester groups formed by the reaction between HTPB and TDI, evidencing urethane bond formation. Additionally, a slight decrease at ~ 3350 cm^{-1} was noted, related to OH consumption from HTPB, concomitant with the appearance of the NH band at ~ 3320 cm^{-1}, indicating a wavenumber shift (Smith 1979).

Association between FT-IR data and viscosity

As previously noted, process control is performed by viscosimetry, which assesses curing in the bulk, and the increase in viscosity is related to reaction progress. It should be emphasized that viscosimetry reflects the behavior of the overall reaction medium, whereas the FT-IR methodology used in this study monitors the reaction specifically at the liner surface. Figure 6 presents a possible relationship between the surface fraction of reacted NCO and the increase in viscosity for R(NCO/OH) = 1.05, an excess considered suitable for FT-IR quantification of NCO decrease due to the lower methodological error and the achieved quantification limit.

According to the curves shown in Fig. 6, a convergent behavior is observed when the curing process is assessed by different methodologies. As the reaction between NCO groups from the curing agent (TDI) and OH groups from the resin (HTPB) proceeds, crosslink formation increases viscosity, since medium resistance rises and fluidity decreases over time. In parallel, the FT-IR methodology, by evaluating only the surface, indicates an increase in the percentage of reacted NCO at the interface, leading both approaches to coherent trends in the respective parameters.

A potential correlation between the curves, and hence between the methodologies, arises from the fact that as NCO groups from the curing agent are consumed, viscosity tends to increase. This effect can be observed, for example, at 0.5, 1.0, and 2.0 h,



Source: Elaborated by the authors.

Figure 6. Relationship between surface consumption of NCO groups and viscosity increase for R(NCO/OH) = 1.05.

where viscosimetric measurements corroborate the determination of reacted NCO, confirming that reaction progress is reflected simultaneously in viscosity increase and in the reduction of NCO available at the surface.

Evaluation of remaining NCO groups on the liner surface

Because FT-IR in reflection mode provides analysis of NCO content at the material surface, the percentage of NCO remaining at the interface (Eq. 5) was considered a relevant assessment parameter. This parameter is directly associated with adhesion, since greater surface availability of NCO contributes to the formation of urethane bonds that are essential for chemical bonding between the surfaces.

$$\text{Percentage of NCO remaining on surface} = 100\% - \%\ \text{NCO reacted} \quad (5)$$

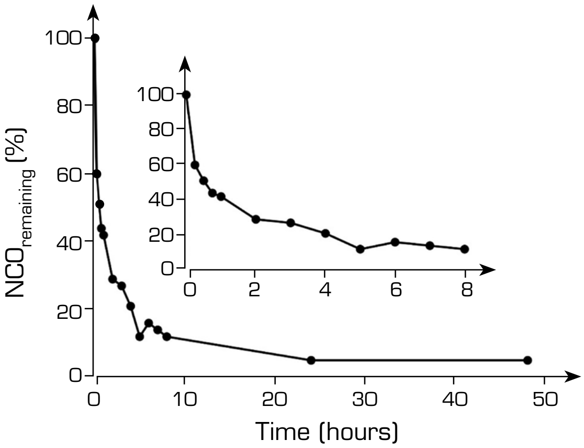
Based on Eq. 5, the corresponding percentages were calculated for each evaluated R(NCO/OH) ratio and are presented in Table 3. The time-dependent variation of these percentages is shown in Fig. 7 for R(NCO/OH) = 1.05, with similar behavior observed for the other ratios.

The behavior observed for the percentages of remaining NCO groups over time, for all evaluated R(NCO/OH) values and across the entire time window considered, suggests a decreasing exponential trend.

Table 3. Percentage of NCO groups remaining on the liner surface for each R(NCO/OH) value.

Time (h)	Percentage of NCO remaining on surface			
	R = 1.00	R = 1.05	R = 1.10	R = 1.15
0.00	100	100	100	100
0.25	60	60	68	70
0.50	51	51	54	65
0.75	43	44	50	59
1.00	39	42	45	52
2.00	24	29	27	38
3.00	20	27	26	28
4.00	15	21	21	29
5.00	14	12	14	21
6.00	10	16	14	21
7.00	8	14	12	21
8.00	7	12	14	16
24.00	2	5	7	7
48.00	4	5	2	4

Source: Elaborated by the authors.



Source: Elaborated by the authors.

Figure 7. Percentage of NCO groups remaining on the surface for R(NCO/OH) = 1.05.

CONCLUSION

Given that the specific objective of this study was to develop an alternative FT-IR/UATR methodology to determine NCO contents at the liner surface, faster and more accurate than the current viscosimetry-based approach, the objective was achieved, as evidenced by the following points:

- The percentage of reacted NCO groups was approximately 80%, with a methodological error of 5%, an acceptable value under the applied analytical conditions and consistent with the 3-7% range reported in FT-IR studies using a similar calculation methodology for aerospace adhesive systems. These results support the effectiveness of FT-IR in reflection mode (UATR) for assessing liner curing.
- Considering that the surface NCO quantification limit is on the order of 5 h for R(NCO/OH) = 1.05, 1.10, and 1.15, and that the lowest methodological error, around 4%, was obtained for R(NCO/OH) = 1.05, this excess is the most appropriate for quantifying NCO decrease by FT-IR. For R(NCO/OH) = 1.15, an even lower error was observed; however, the mechanical properties of the liner should be evaluated, since a higher amount of curing agent can alter those properties.
- Regarding the association between FT-IR and viscosimetry data, establishing the parameter "percentage of NCO remaining at the surface" was relevant to create references for determining the tack point between methodologies. Therefore, the proposed approach is appropriate for its intended purpose, offering a faster and more precise procedure (4% error for R(NCO/OH) = 1.05) than viscosimetry for determining the tack point, whereas viscosimetry adopts a 10% deviation margin relative to the mean curve.

From an application perspective, the proposed FT-IR/UATR methodology can be integrated into the rocket motor casting process as a real-time analytical tool to determine the ideal tack point with greater precision and reproducibility. By replacing empirical evaluations with quantifiable chemical parameters, this approach enhances process reliability and minimizes the risk of adhesive failures, ensuring the structural integrity of rocket engines during subsequent preparation and operational stages. Therefore, its implementation represents a significant technological advancement toward a more reliable, controlled, and predictable casting process for solid propellant rocket motors.

The use of NIR is less frequent than in the MIR region. When applied, including in reaction studies, it is predominantly employed in transmission mode, the more conventional approach (Romão *et al.* 2004). Therefore, an opportunity for continuing this study is to assess liner curing by reflectance analysis in the NIR region, that is, by NIRA, a technique that has been used recently (Passero *et al.* 2024) with success in other aerospace systems, including materials for flexible thermal protection.

CONFLICT OF INTEREST

Nothing to declare.

AUTHOR CONTRIBUTIONS

Conceptualization: Rios RM, Dutra RCL, and Pinto JRA; **Methodology:** Rios RM, Dutra RCL, and Pinto JRA; **Investigation:** Rios RM, Diniz MF, and Santos RS; **Writing – Original Draft:** Rios RM; **Writing – Review & Editing**: Rios RM, Dutra RCL, Sanches NB, and Pinto JRA; **Funding Acquisition:** Dutra RCL; **Resources:** Santos RS and Pinto JRA; **Supervision:** Dutra RCL, Sanches NB, and Pinto JRA; **Final approval:** Dutra RCL.

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All data sets were generated or analyzed in the current study.

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DECLARATION OF USE OF ARTIFICIAL INTELLIGENCE TOOLS

The authors declare that no artificial intelligence tools were used in the preparation, writing, data analysis, or review of this manuscript.

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