CAAP Quarterly Report

October 25, 2025

Project Name:
Selection and Development of Safer Polymer and Composite Pipeline Liners through Microstructural and Macroscopic Study of Materials and Designs

Contract Number: 693JK32250001CAAP

Prime University: Brown University

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Reporting Period: Q10

Project Activities for Reporting Period

A paper titled "Environmental aging of polymers to evaluate their potential for remediating natural gas pipelines," that was an outcome of this research went through peer-review process and was published in the journal of *Industrial & Engineering Chemistry Research*, during this quarter. The full information for the paper is as follows:

- Hajirezaei, M., Ferreira, A., Campbell, M., Ghosh, D., Alkhoury, K., Lyakhovych, Z., Mathiowitz, E., Srivastava, V., and Poling-Skutvik, R., "Environmental aging of polymers to evaluate their potential for remediating natural gas pipelines," *Industrial & Engineering Chemistry Research* 64 (36), 17724-17731, 2025

During this quarter, in Poling-Skutvik's laboratory, we continued our accelerated aging experiments by exposing additional polymer samples to the NG2 gas mixture (containing oxygen contaminants) at both elevated temperature (90 °C) and room temperature. In earlier studies, we found that the activation energy under NG2 decreases much more rapidly than under NG1, reaching its minimum after just 7 days of exposure, whereas a similar reduction required approximately 30 days under NG1. Beyond this point, the activation energy values plateau, indicating that the degradation process approaches a steady state relatively quickly in the presence of oxygen. Figure **??** shows the results for the samples that were exposed to NG2 at both high temperature and room temperature. As shown in this figure, the activation energy exhibits a larger decrease under the high-temperature condition compared to room temperature, indicating that thermal acceleration significantly enhances the degradation processes occurring in the polymer.

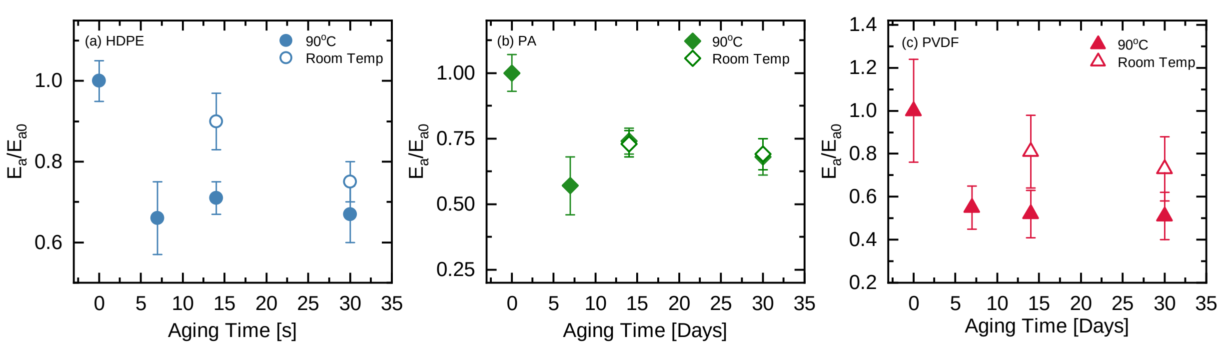
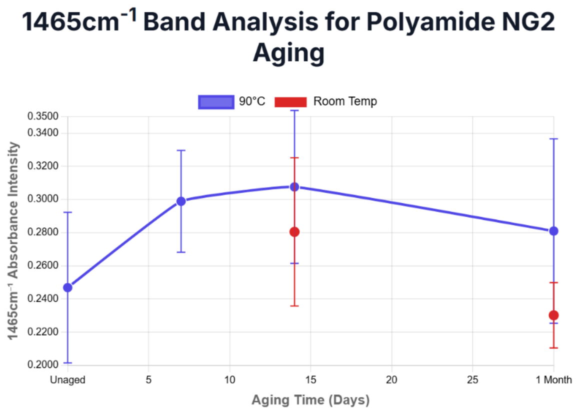


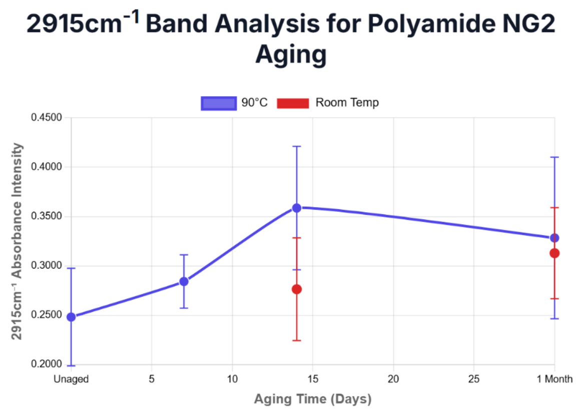
Figure 1: Normalized activation energy $E_a/E_{a,0}$ for (a) HDPE, (b) PA and (c) PVDF.

In this quarter, in the Mathiowitz lab, most of the NG2 characterization testing was completed. For polyamide samples, we consistently saw greater intensity for samples that have been aged. The average intensity at 2915 cm^{-1} and 1465 cm^{-1} samples increased with aging time, with moderate to high variation across samples (Figure 2a and 2b). Samples that were aged at room temperature consistently showed lower intensities than samples aged at 90°C. As can be seen, neat polyamide has an inherent intensity in these regions, so sample-to-sample variation is expected. PVDF displayed a similar trend with all aged samples showing higher intensities at 1465 cm^{-1} and 2915 cm^{-1} than neat samples (Figure 2b and 2d). At

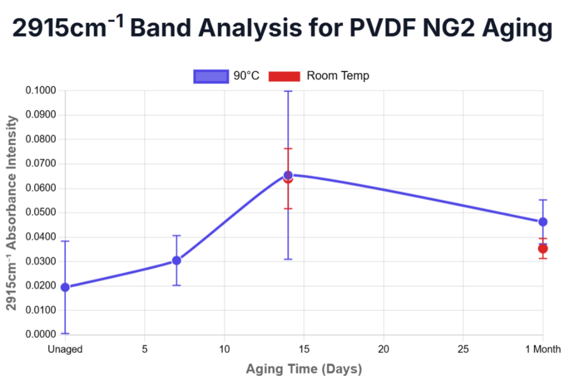
2 weeks, there are some inconsistencies with high variation, and the 2915 cm^{-1} band shows higher intensity after 2 weeks than 1 month, as well as increased intensity at 2 weeks Room Temp. The overall intensity at both of these bands is low.



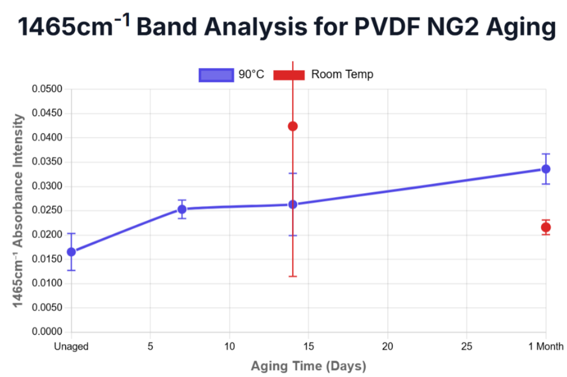
(a) Intensity of 1465 cm^{-1} band for PA NG2.



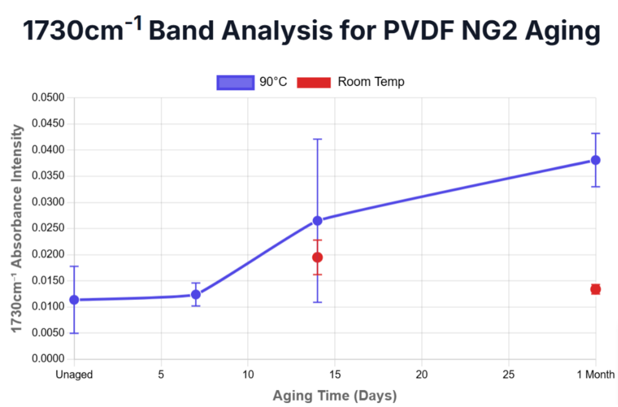
(b) Intensity of 2915 cm^{-1} band for PA NG2.



(c) Intensity of 2915 cm^{-1} band for PVDF NG2.



(d) Intensity of 1465 cm^{-1} band for PVDF NG2.



(e) Intensity of 1730 cm^{-1} carbonyl band for PVDF NG2.

Figure 2: FTIR band intensities for Polyamide (PA) and PVDF NG2 samples.

With the addition of the oxygen component to NG2, we additionally investigated if any degradation indicators would appear on the FTIR spectra, such as the carbonyl band at 1730 cm^{-1}. There were changes in the intensity in this band that can most clearly be seen in the PVDF, with the 90°C temperature clearly contributing to a higher intensity, with nearly no increase occurring at Room Temp (Figure 2e). Changes in the intensity in this region can be seen in some HDPE and Polyamide samples, but the trend is not consistent.

In this quarter, in the Srivastava lab we completed the mechanical testing on polymer samples exposed to the NG2 gas mixture at room temperature for durations of 14 and 30 days. We did not observe any significant changes in the deformation response of HDPE (Figure 3a, 3b, and 3c) and PVDF (Figure 3g, 3h, and 3i) across the three different strain rates of $0.0005s^{-1}$, $0.005s^{-1}$, and $0.05s^{-1}$, and across different aging duration and conditions (aging at 90°C versus at room temperature). Thus, suggesting that exposure under elevated temperature does not change the constitutive mechanical response of these two materials. On the other hand, we observe a significant reduction in the peak stress of PA material and its overall deformation mechanical response for samples aged at room temperature, compared to the ones exposed at 90°C (Figure 3d, 3e, and 3f).

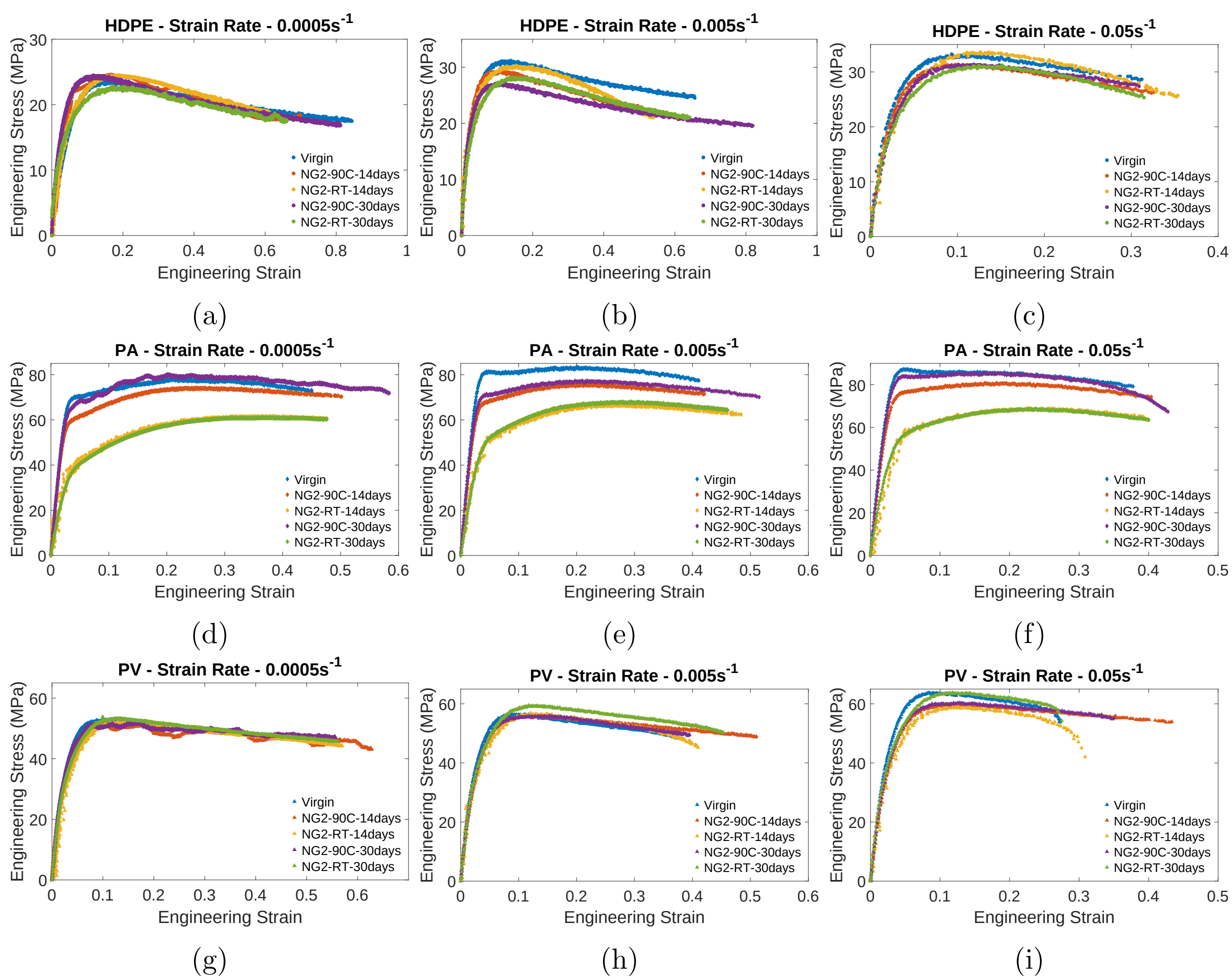


Figure 3: Stress-strain response of (a-c) HDPE, (d-f) PA, and (g-i) PVDF at strain rates of $0.0005s^{-1}$, $0.005s^{-1}$, and $0.05s^{-1}$, respectively.

Project Financial Activities Incurred during the Reporting Period

Costs associated with PI partial summer support, PhD graduate student support, equipment operations, shared facility use, and materials and supplies for experimental research work for the project were supported.

Table 1 Summary of Q10 DOT Funded Spending Annual Report-07/01/2025-09/30/2025				
Institution		Amount ($)	Amount ($)	Subtotal ($)
Brown University	Category	Salary ($)	Fringe ($)	
	PI	4,863.72	1,459.11	6,322.83
	Co-PI	5,864.04	1,759.21	7,623.25
	Postdoc			-
	Graduate Students	22,390.34	1,162.60	23,552.94
	Undergraduate Students			-
	Graduate Student Fees			34,510.18
	Facility Usage			2,167.50
	Purchased Services			-
	Materials and Supplies			511.16
	Travel			-
	Equipment			-
	Total Direct			74,687.86
	Indirect			23,905.77
	Subtotal			**98,593.63**
University of Rhode Island	Personnel	Salary ($)	Fringe ($)	
	Salaries	33,747.81	316.66	34,064.47
	Operating Expenditures			4,313.88
	Travel			-
	Student Aid			6,310.00
	Total Direct			44,688.35
	Indirect			22,067.57
	Subtotal			**66,755.92**
Total				**165,349.55**

Project Activities with Cost Share Partners

Partial support for graduate students has been continuously provided during the academic years as per the cost-share agreement.

Project Activities with External Partners

The PI and Co-PI from Brown University met with the sub-awardee University of Rhode Island (URI) researchers on bi weekly basis this quarter to share the research results, discuss the outcomes and decide and plan future research steps. The team from URI and Brown University shared polymer samples for collaborative testing and characterization.

Potential Project Risks

As the research progresses and more experimental data is collected, since the work and findings for liner polymer materials are new, there could be a risk of unanticipated new findings. This risk is and will be managed by adjusting the research methods as new data comes. Inconsistencies/microscopic heterogeneity across individual samples may make morphological conclusions for macroscopic samples difficult.

Future Project Work

Our future work will focus on continuing to test our samples after exposure to more corrosive gases, such as H_2S and BTEX.

Potential Impacts to Pipeline Safety

Previous testing established changes in samples that were established by x-ray diffraction. This method is impractical in the field with XRD machines being bulky and immobile. If similar measurements can be made with an FTIR, which is a significantly more mobile tool, we can detect phase content changes that may be precursors to mechanical failure in the field. This new consideration of testing materials for potential problems can be valuable. The experimental methods being evaluated, the experimental data that is being measured and collected and fundamental understanding of liner polymer materials' response, materials properties, and safer liner material guidance obtained through this collaborative research will help increase the understanding and safety of polymer liners for pipelines.

Supplement

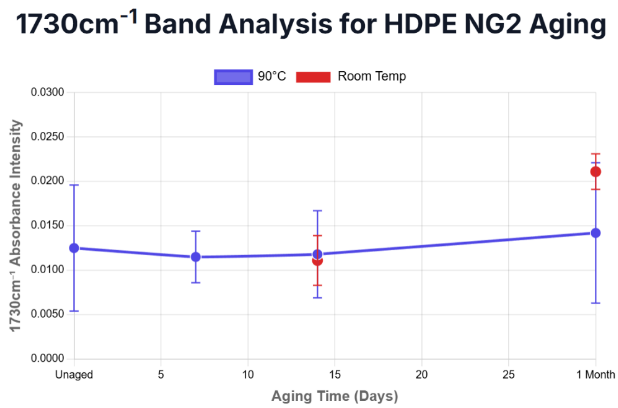


Figure 4: Carbonyl band intensity for HDPE

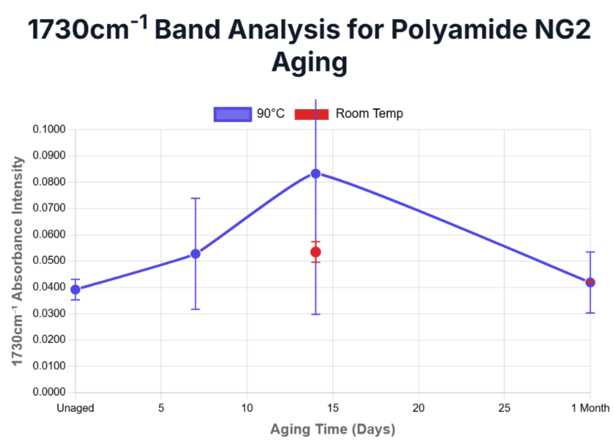


Figure 5: Carbonyl band intensity for Polyamide