Chemicals Aging and Degradation Mechanism of SLA Printed Materials for Nuclear Energy Applications

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- Joseph Munoz, UG RA
  - **Previous Expertise**: Material characterization in harsh environments, DIW

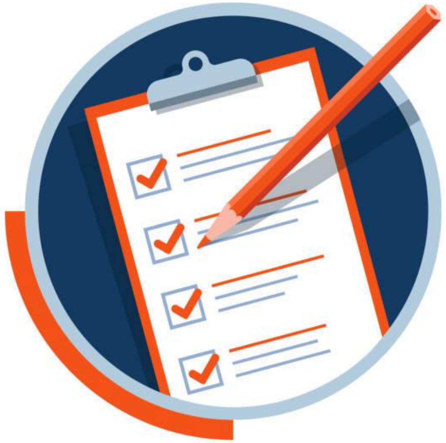
- Joshua Dantzler, PhD RA
  - **Previous Expertise:** Ceramic photopolymer resin formulation & printing, DIW printing of ceramics, lattice structure design

- Md Sahid Hassan, PhD RA
  - **Previous Expertise**: VP printing of polymers, PBF printing of composites, material testing & characterization

- Md Shahjahan Mahmud, PhD RA
  - **Previous Expertise**: SLA and DLP printing of polymers, material testing & characterization

Overview

- Project Objectives
- Task Description
- Parameters
- Task Updates (Work done so far)
- Future Plans
- Q&A



My Background

As an Undergraduate student attending the University of Puerto Rico – Mayagüez Campus, I am deeply immersed in the field of Electrical Engineering. My expertise and prior experiences revolve around unmanned systems, autonomous robotic operation, FDM printing, and control systems. During my academic journey, I have collaborated actively in a project centered on chemical aging and characterization tests with polymer materials. This opportunity has further expanded my knowledge and understanding of materials science and engineering applications. I am passionate about exploring innovative solutions to real-world challenges and look forward to contributing my skills and knowledge to the advancement of cutting-edge technologies in my chosen field.

Objectives:

- Printing & testing samples using commercial photopolymer resin
- Investigating effect of print orientation on material properties
- Aging of specimens in chemical environments
- Characterization of material properties before & after aging (Stress, strength, swelling, leaching, FTIR)
- Study and analysis of aging mechanism & acceleration

Materials:

- **Resin:** Formlabs V4 clear resin
- **Chemicals/Solvents:**
  - 6M HCl
  - 6M HNO3
  - 10% Acetone
  - Xylene
  - Dodecane
- **Printer:** Formlabs Form3 SLA Printer
- **Washing device:** FormWash
- **Curing device:** FormCure

Clear resin ingredients:
Urethane Dimethacrylate(UDMA): 55-75%
Methacrylate: 15-25%
Photoinitiator: <0.9

Formlabs Form3 SLA Printer

V4 Clear resin

FormWash

FormCure

Task Description

Part-1: Printing samples with different print orientations	➢Printing test samples at different orientations, such as: • Tensile sample printing at X, Y & Z orientations • Compression sample printing at 0, 45 & 90◦ angles • Harness Test sample printing at 0, 45 & 90◦ angles ➢Material property analysis for each orientation
Part-2: Printing test samples & Chemical aging	➢Printing test samples ➢Aging of samples in chemical environments • At different exposure time • At different temperature
Part-3: Material Characterization	➢Performing mechanical, chemical & characterization tests (Swelling, Leaching, Tensile, Compression, SEM, FTIR)
Part-4: Data Analysis & Final Report	➢Analysis of aging mechanism & acceleration from the experimental data ➢Final report preparation

Instron machine

Thermo FTIR Nicolet IS5

Parameters:

Printing Parameters: [1, 2, 3]

Parameters	Quantity
Printer Laser spot size	85um
Laser wavelength	405nm
Layer Height	100um

Post Processing parameters: [2,3]

Process	Quantity
Washing time	15 min
Curing time	60 min
Curing temperature	60◦C

Reference:

[1] https://formlabs.com/3d-printers/form-3/tech-specs/
[2] formlabs-materials-library
[3] https://support.formlabs.com/

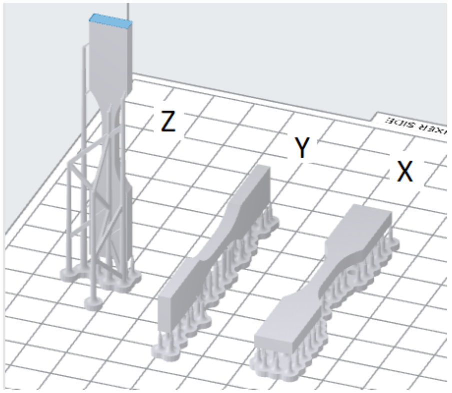
Aging parameters:

Duration of Chemical exposure (weeks)	1	3	6	9	12
Aging Temp.	Room Temperature				
Chemicals	6M HCl, 6M HNO3, 10% Acetone, Xylene & Dodecane				

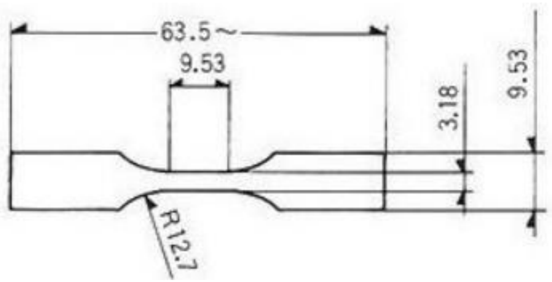
Chemical exposure

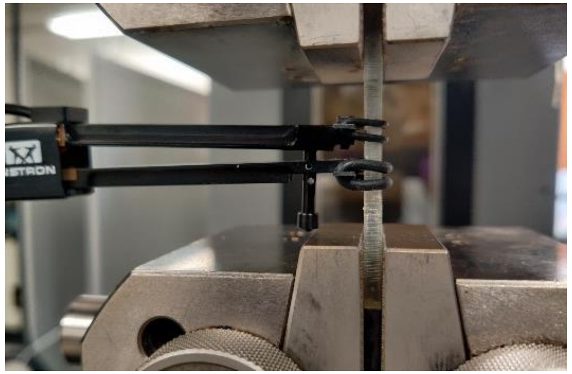
Task: Part 1 (Print Orientations)

❖ Tensile Test: ASTM D638



Print orientation	Elongation (%)	Ultimate Stress (MPa)	Young's Modulus (GPa)
Formlabs data [2]	**6.0**	**65.0**	**2.80**
X	5.5	69.8	2.74
Y	7.0	67.1	1.96
Z	6.5	82.4	2.81





Reference:

[2] formlabs-materials-library

Test machine	Instron (50KN)
Disp. rate	1mm/min

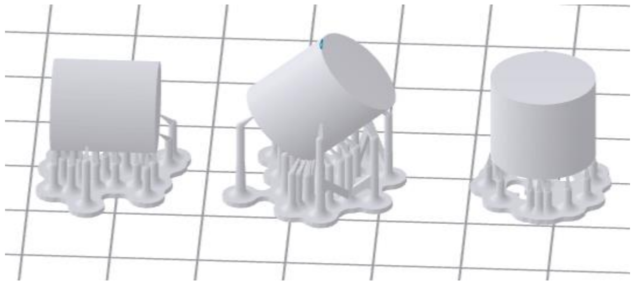
Task: Part 1 (Print Orientations)

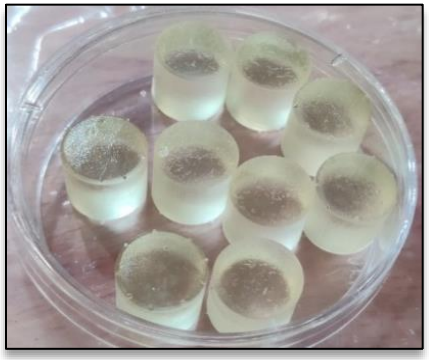
❖ Compression Test: ASTM D695- 2015

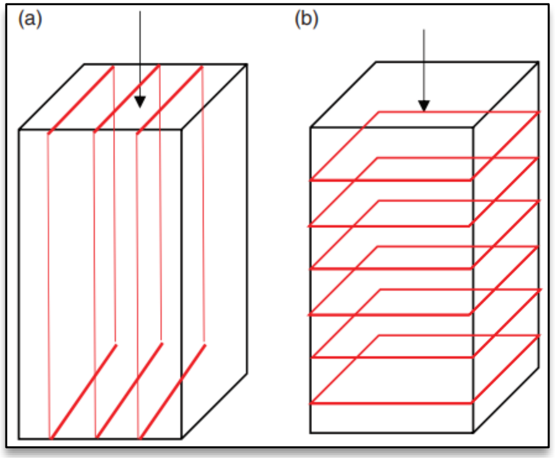
Print Orientation	Yield stress, MPa	Max Compressive strength, MPa	Young's Modulus, GPa
0◦	111.0	263.0	2.05
45◦	101.2	313.5	1.75
90◦	97.5	315.3	1.60

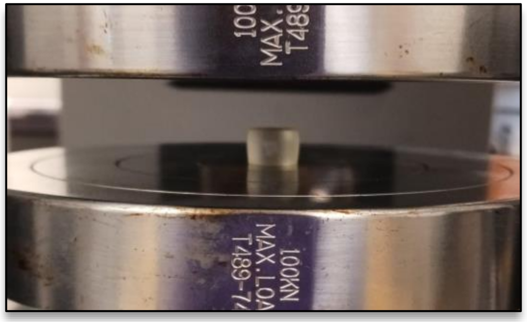
Test machine	Instron (50KN)
Disp. rate	1mm/min

Sample Dimension
(ΦxH): 12x12 mm









Task: Part 2 (Chemical Aging Schedule)

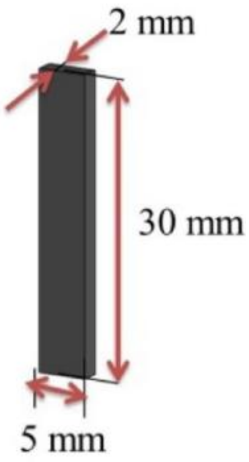
Test	Solutions/Solvents	W1	W3	W6	W9	W12
Swelling	HNO3					
	HCl					
	Dodecane					
	Xylene					
	10% Acetone					
Compression	HNO3					
	HCl					
	Dodecane					
	Xylene					
	10% Acetone					
Tensile	HNO3					
	HCl					
	Dodecane					
	Xylene					
	10% Acetone					

Room Temperature Test

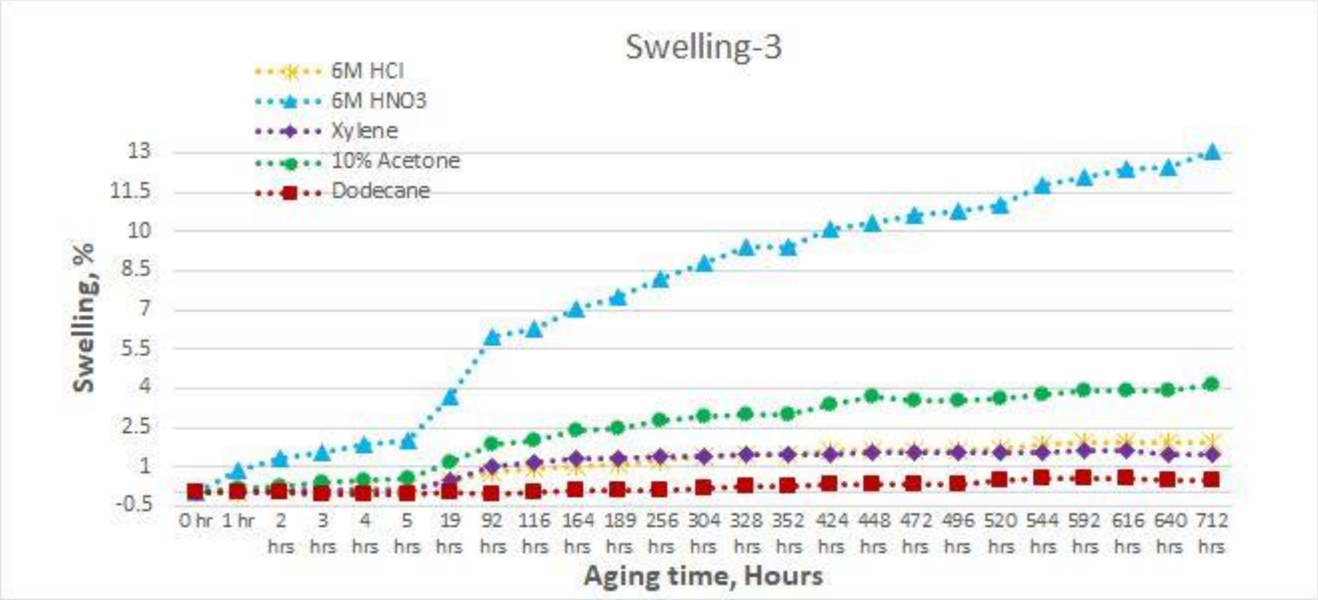
Swelling Test:

Weight Scale
(Fisher Scientific)

Sample Dimension
(WxHxt): 05x30x02mm

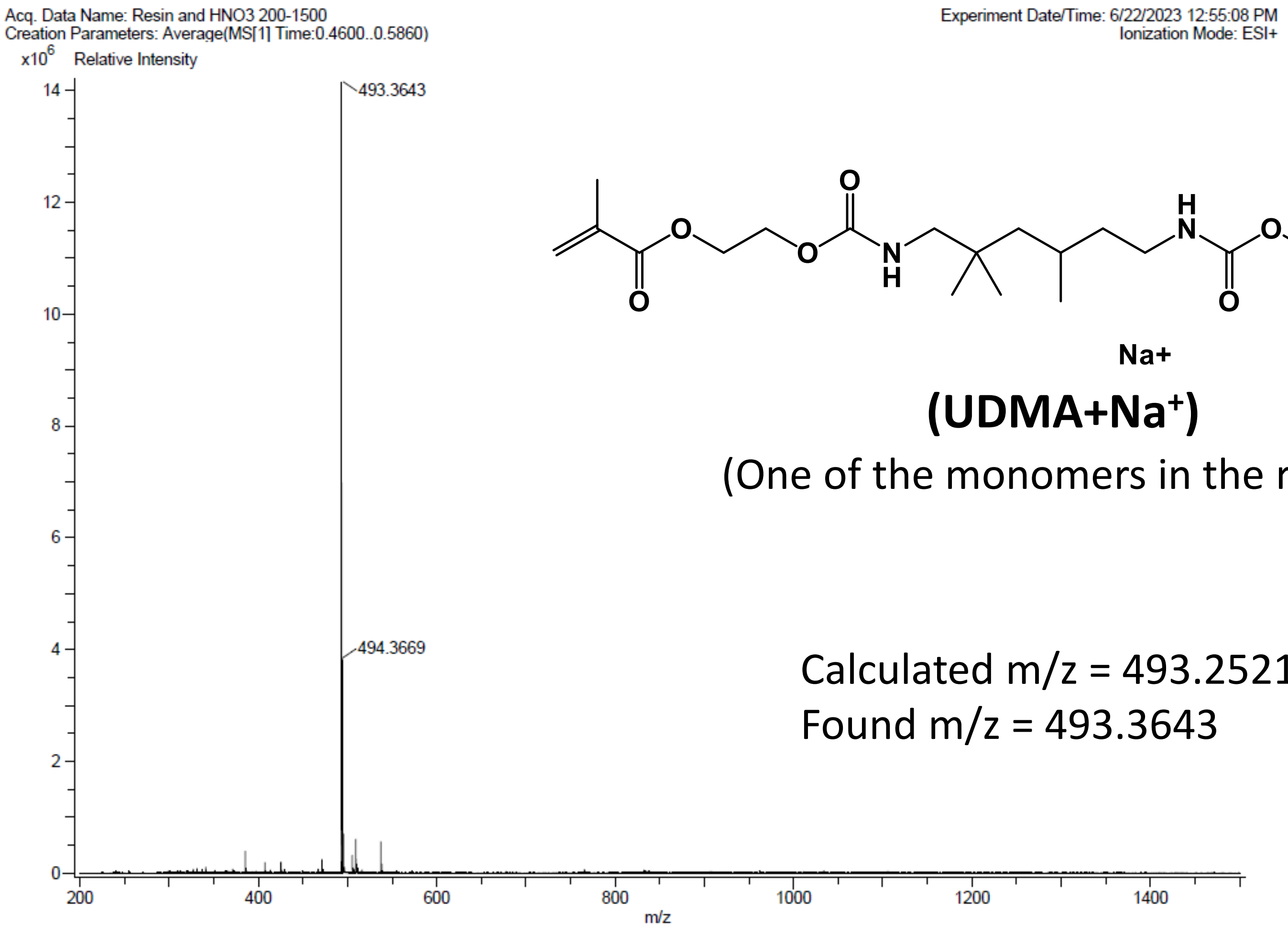


Aging of Swelling sample



Leaching Test: Mass Spectrometry

Formlab V4 Clear Resin



(UDMA+Na^+)

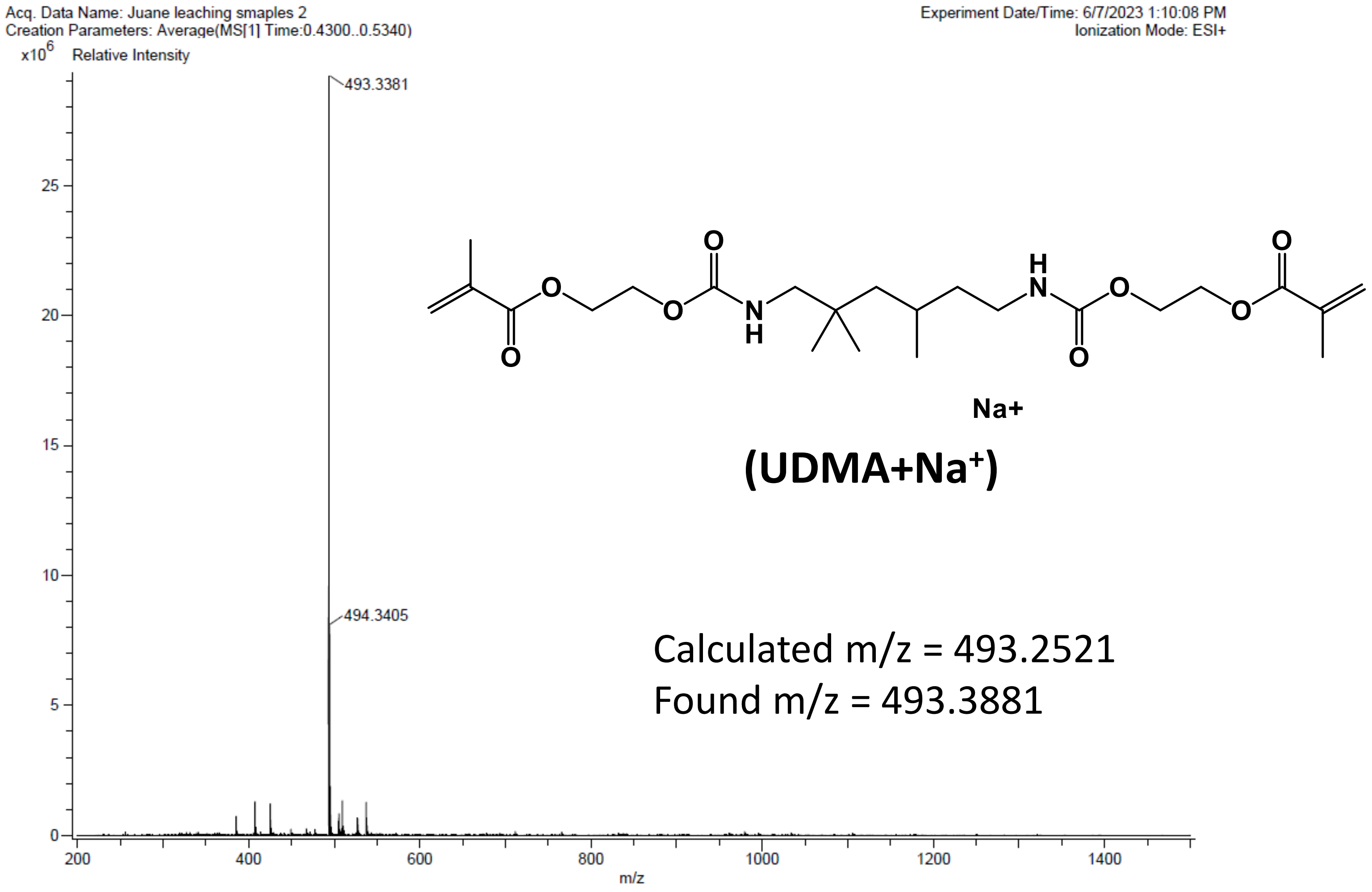
(One of the monomers in the resin)

Calculated m/z = 493.2521

Found m/z = 493.3643

Leaching Test: Mass Spectroscopy

Solution from Xylene



(UDMA+Na$^+$)

Calculated m/z = 493.2521
Found m/z = 493.3881

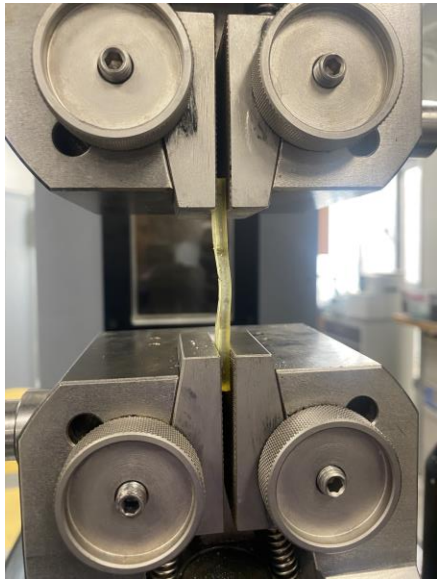
Leaching Test: Mass Spectroscopy

Leaching Test Summary:

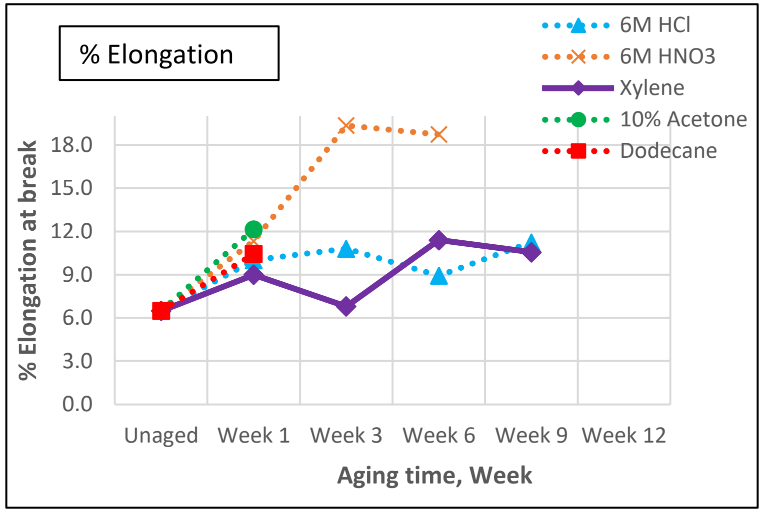
Experiment	Exposure time (days)	Detection	Conclusion
Xylene	41	**($UDMA+Na^+$)**	Leached out
10 % Acetone in Water	30	**($UDMA+Na^+$)**	Leached out
Dodecane	30	**($UDMA+Na^+$)**	Leached out
HNO_3 6 M	30	Peaks are present in commercial acid as well.	Analysis ongoing
HCl 6M	41	Peaks are present in commercial acid as well.	Analysis ongoing

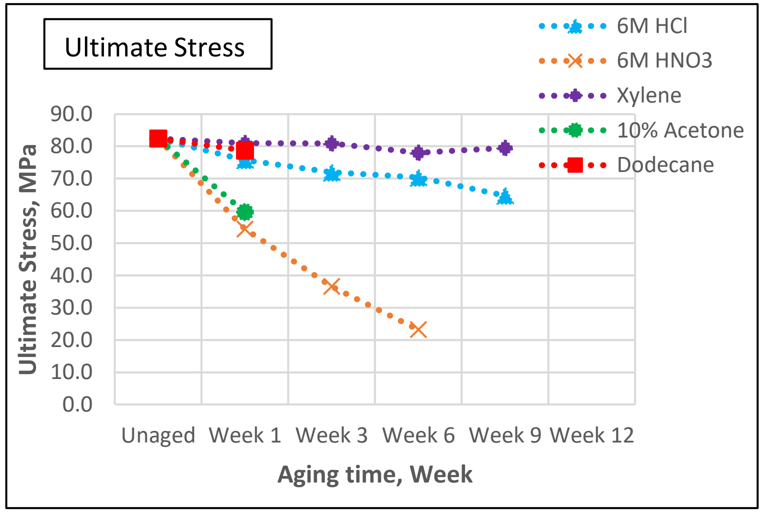
❖Tensile Aging Test:

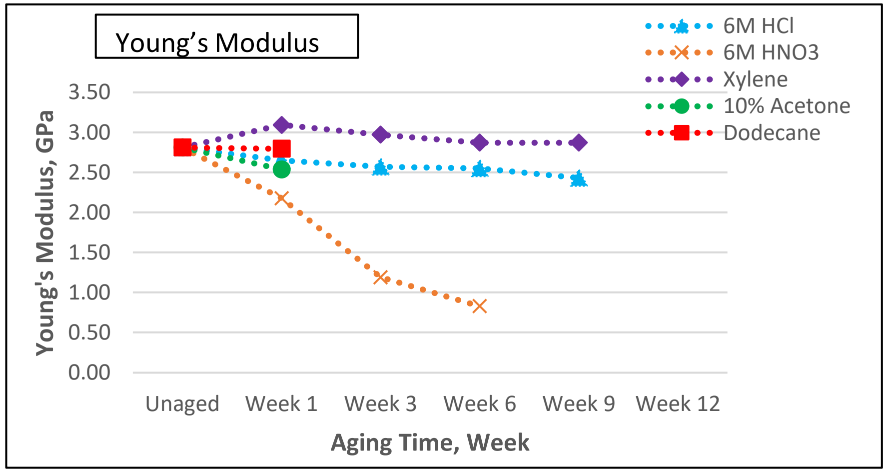
Tensile Test comparison before & after aging, Z orientation:



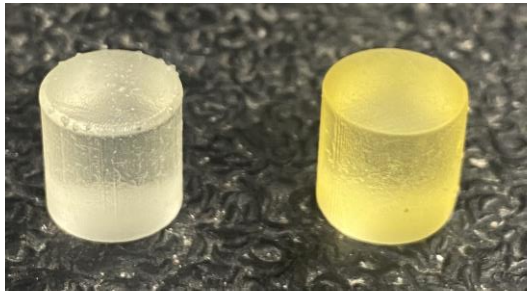
Week 9 Tensile HNO3







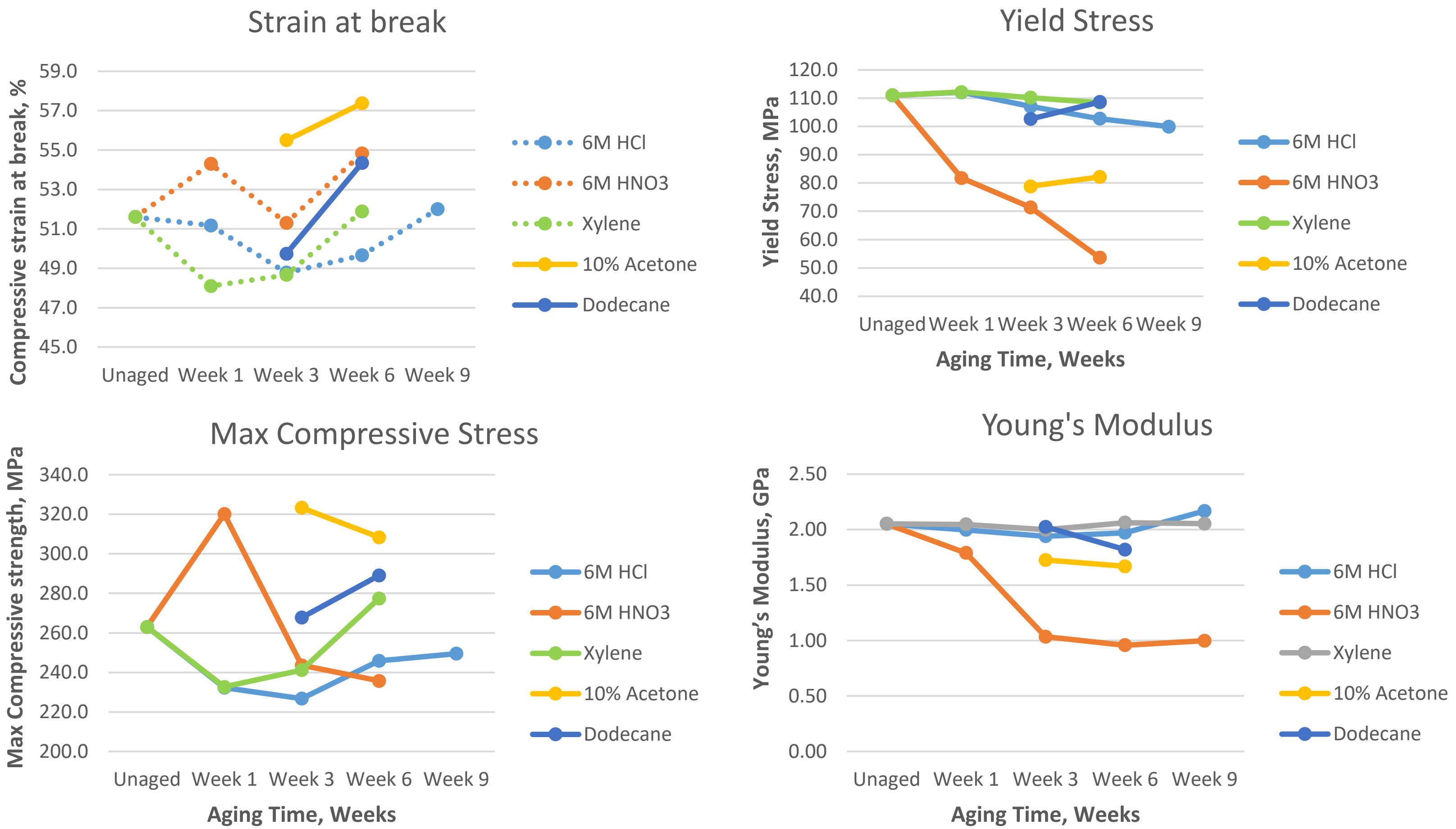
❖ Compression Aging Test:



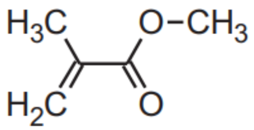
Unaged vs week 9 for HNO3

HNO3 Aging Sample from W1 to W9

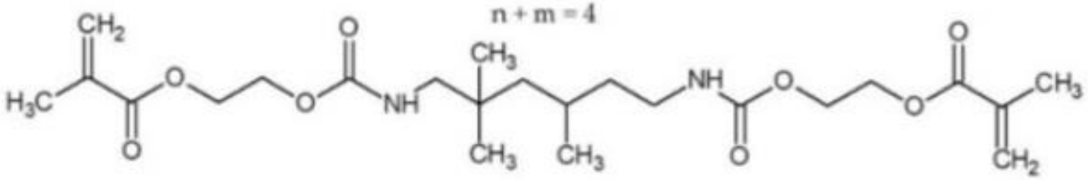
Compression Test Comparison before & after aging: Week-6



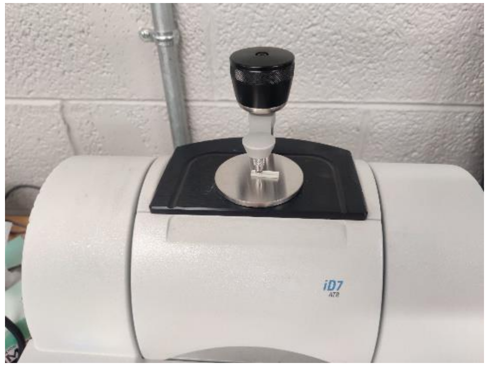
FTIR Spectra: After aging (Week-12)



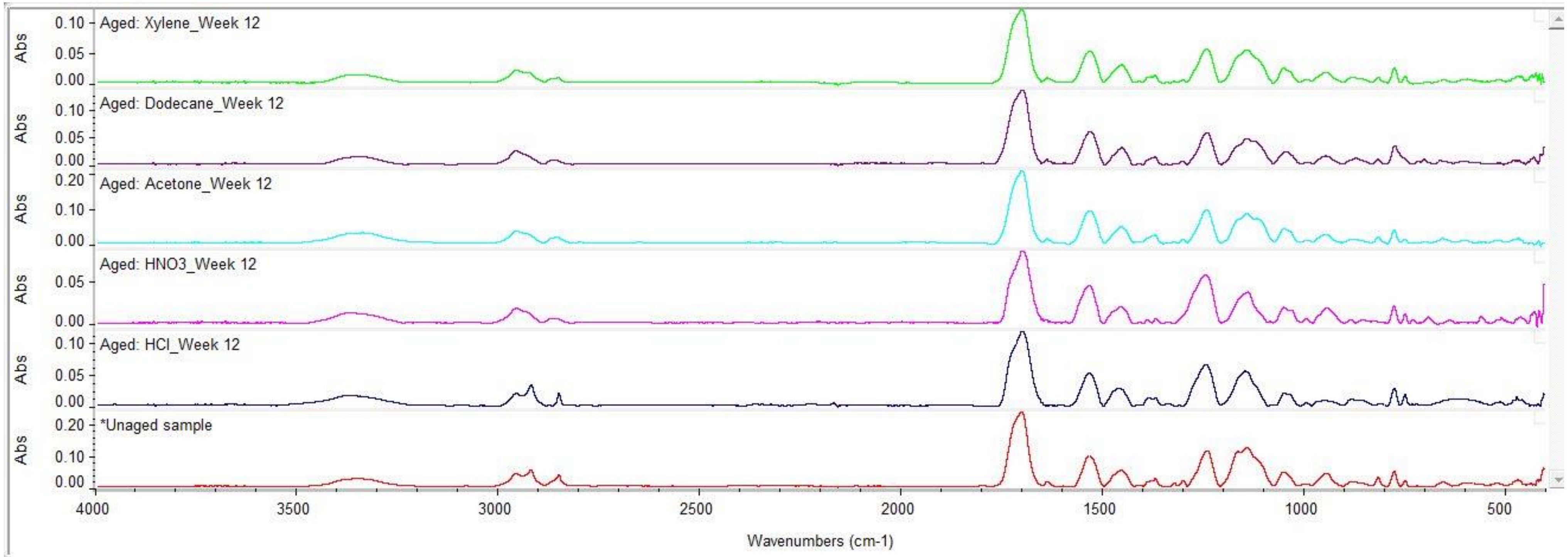
Methyl Methacrylate (**MMA**)



Urethane di-methacrylate (**UDMA**)



Thermo FTIR Nicolet iS5



3340: N-H group
2918/2850: Methyl- C-H stretching vibration
1700: C=O stretching
1539/1452: Methyl- C-H bending vibration
1297: C-O stretching
944: Olefin group; C-H bending vibration (occurred due to oxidation)
815: C=C bending vibration (Methacrylate double bond)

850-550: C-Cl stretching

Future Work Plan

- Finish Room Temperature Tests
- Analyze Final Data and final report
- Begin Thermal Chemical Aging testing
- Research Post-Printing Improvement on Commercial Resin for Better Mechanical Properties

Thank You!

Questions?

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