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Protocols for the Environmental Degradation of Polymers using Raman Spectroscopy

Ashlyn Lee, Western Washington University, Department of Environmental Sciences,

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This quarter I had the opportunity to work in Dr. Montaño's laboratory to develop protocols studying the environmental degradation of polymers using Raman Spectroscopy. We chose to use the Raman Microscope for this experiment because it is often used to detect chemical structures by laser excitation, therefore we could use this technique to visually understand the changes in structure made to the test material before and after experimental weathering. To conduct this experiment, I chose two environmentally relevant polymers: Low Density Polyethylene (LDPE) and Polycarbonate (PC). These polymers are used in a wide variety of plastic packaging and additives commonly found in microplastics. To simulate environmental stressors, we chose to chemically degrade LDPE and PC using hydrolysis and photolysis. Hydrolysis is the chemical degradation of materials through reactions with water and photolysis is the chemical degradation of materials through reactions to ultraviolet (UV) light.

The presence, degradation, and bioaccumulation of microplastics is a serious threat to environmental and human health, more recent studies have found microplastics in placenta and lung tissue (Jenner et al., 2022). It is important to understand the chemical changes made to these polymers by environmental degradation so we can further investigate their bioaccumulation properties, fate and transport, and leachate concentrations. The objective of this study was to use Raman spectroscopy to map the changes in photon scattering of two polymers after weathering to understand how their surface chemistry changes as a result of environmental degradation.

The experimental procedure was adopted from previous studies from Harraq et al, 2022 and Sun et al., 2020 documenting weathering methods on polymers to assess degradation. I started by making solutions of saltwater for the hydrolysis testing into four treatment groups with triplicates: 0 g/L, 2 g/L, 6 g/L, and 12 g/L. These were the concentration gradients chosen because they represent a gradient that is similar to marine concentrations. To prepare the test materials, I cut the polymers into evenly sized slices, both polymers were weathered with the chosen concentration gradient in simulation vials. The simulation vials were randomly assorted into a vial rack and placed outside on the roof of the Environmental Studies building at Western Washington University for 48 hours for photolysis exposure. After the 48 hour exposure period the samples were collected and placed into the laboratory refrigerator for another 7 days.

For the analysis, we chose to use Raman Spectroscopy to analyze the surface chemistry changes to the polymers to understand how hydrolysis and photolysis will affect the functional group structure of these compounds. Using the excitation of photons to evaluate the shifts of the polymers after weathering has very important implications to microplastic pollution, degradation, and uptake in the environment. The samples were cleaned prior to analysis and viewed under the 532 nm laser for optimal scattering. After analyzing, the Raman shifts and normalized intensity for both polymers was plotted with the highest salinity treatment against the control to visualize the effects of the hydrolysis testing. The untreated (control) polycarbonate and the treated (12 g/L) polycarbonate samples were a 93% match according to the Raman spectra search. The shifts were generally matched however the treated polycarbonate showed a few shifts of greater intensity that the untreated sample did not (Figure 1).

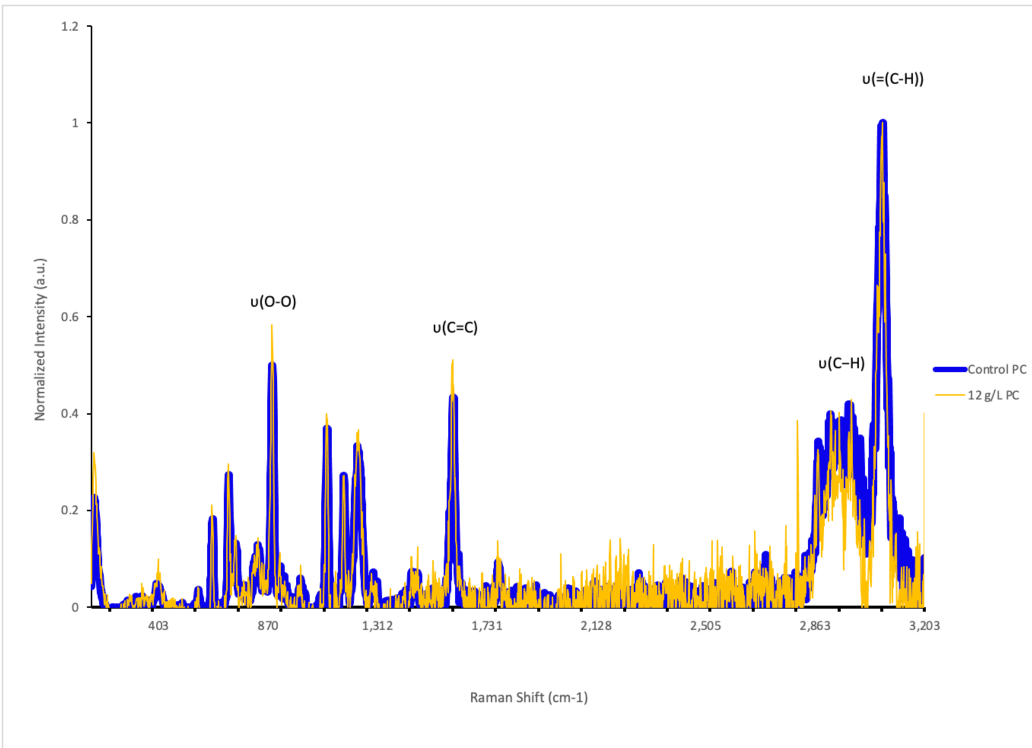

Figure 1. Raman shift (cm^{-1}) against normalized intensity of untreated (control) polycarbonate and treated (12g/L) $NaCl_{(aq)}$ polycarbonate using Raman Spectroscopy.

The shifts between the control sample and the highest treated sample for LDPE are visually similar. The intensity and peaks are the same for both samples (Figure 2). The LDPE samples were more similar showing a 95% match between the highest treatment group and the control. The peaks for untreated LDPE and treated LDPE were more similar than for the PC treatment.

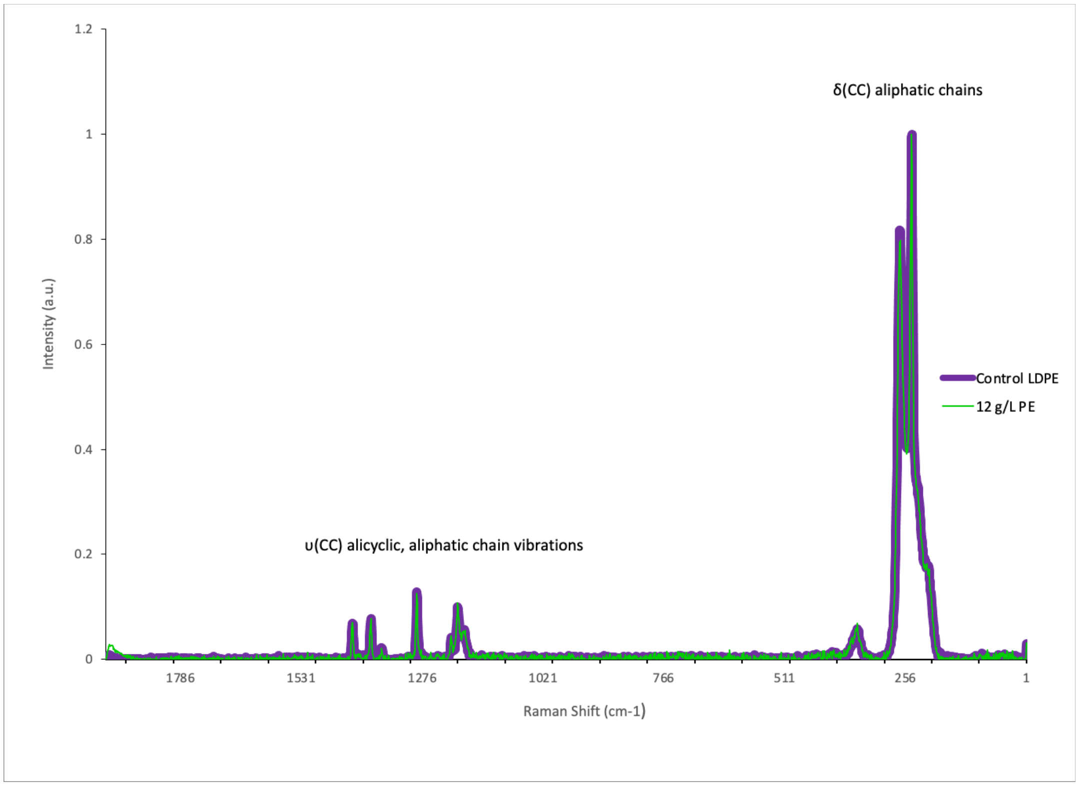

Figure 2. Raman shift (cm^{-1}) against normalized intensity of untreated (control) LDPE and treated (12g/L) $NaCl_{(aq)}$ LDPE using Raman Spectroscopy.

The analysis indicates that there is some change to polycarbonate as a result of environmental degradation (hydrolysis and photolysis) as seen from Figure 1. Further analysis may be done to determine the potential changes to LDPE. Further studies may consider expanding on this experimentation by extending the exposure time between the salinity and UV treatments or quantifying leachate concentrations using QTOF-MS instrumentation.

This opportunity allowed me to design, research and conduct an experiment of my own under the guidance of Dr. Montaño. I read the most recent peer reviewed literature and used my own background knowledge on the subject to submit an experimental proposal and procedure that was reviewed and accepted. Throughout the duration of this experiment, I have learned about laboratory safety, technique and instrument handling. I was able to work independently setting up, calibrating, and running samples on the Raman Microscope. I was challenged and uplifted academically to a standard I have not yet been asked to reach and it was an incredibly rewarding experience. I am very interested in pursuing a masters degree in toxicology and I felt that additional laboratory and research experience was necessary for me.

The experience in both experimental design, proposal writing, and research had developed my academic and professional skills insurmountably. I feel extremely prepared to meet expectations of both professors and colleagues because of this experience. I have a fundamental understanding of laboratory work and I feel much more confident in my abilities to learn new techniques and instrumentation. My confidence as a student and as a scientist has grown throughout this quarter and I am excited to continue my research and laboratory experience.

My education through Western Washington University was valuable to my ability to pursue higher education and experience in undergraduate research. The laboratory classes I took were thoughtful and built upon each other in a way that made me more confident in each lab I stepped into. The assignments and papers I wrote in my toxicology classes were applicable to what I would be expected to do in the professional world. My mentors and professors at Western Washington University inspired me to pursue higher education and a career in the toxicology field. I feel honored to have received the opportunities and accolades I have during my time at this university and I hope to continue this work within the coming years. I want to thank Dr. Montaño for allowing me this experience to work in his laboratory and develop fundamental skills I will need for graduate school and beyond. This internship was extremely important to me and it is something I will reflect on throughout my academic and professional career.

References

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