NM WRRI Student Water Research Grant Progress Report
Final Progress Report

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Title of Project: Evaluation of Heavy Metal Adsorption onto Microplastics

Research Problem and Research Objectives:

Microplastics are ubiquitous pollutants across aquatic ecosystems. However, the occurrence of microplastics in freshwater systems is not well-established compared to the marine environment.[1] Microplastics are broadly defined as plastic materials with a diameter <5 mm.[1] Microplastics with a diameter <150 μm are of particular concern given their small size and their potential to be ingested.[2] The association of microplastics with other contaminants such as heavy metals is potential harmful to humans and the environment. Freshwater systems in the state of New Mexico near abandoned mine sites have concentrations of heavy metals above the EPA maximum contaminant levels.[3] Microplastics introduced into these freshwater systems by recreational activities, solid waste dumping, wastewater treatment effluents, or other sources may interact with heavy metals.[1,4,5] This study is designed to identify the occurrence of microplastics in freshwater systems in New Mexico and to evaluate the adsorption of heavy metals onto them.

The research objectives are:

1. To identify the presence of microplastics in freshwater systems containing elevated concentrations of heavy metals specifically at Tingley Beach, the Rio Grande, and Laguna Pueblo, NM.
2. To evaluate the adsorption of heavy metals onto microplastics in laboratory experiments.
3. To investigate the behavior/mechanism of heavy metal adsorption onto microplastics in freshwater systems and laboratory conditions.

Research Methodology

Figure 1: Sampling locations at (a) Tingley Beach, (b) Rio Grande (North), (c) Rio Grande (Albuquerque), and (d) Laguna Pueblo New Mexico.

Sampling methodology. Two 1L samples were collected from six locations along the Paguate River and freshwater reservoirs near the Jackpile mine of Laguna Pueblo NM, as well as from the Rio Grande, and Tingley Beach in Albuquerque, NM (**Figure 1a-d**). The Albuquerque locations assist in the analysis of microplastic occurrence in freshwater systems used for recreational activities. Field controls were included when sampling. Samples were taken with one (1) L borosilicate glass bottles previously cleaned by sonicating in a Cole-Parmer CPXH Series Sonicator with ultra-pure water (18 MΩ) for 30 min. After sampling, all bottles were capped with aluminum foil to prevent airborne interference. Samples were placed in a cooler until their storage at 4°C. All the experimental apparatus was sonicated for 30 min in an ultrasonic bath with ultra-pure water (18 MΩ) and cover with aluminum foil before use. The sonication was performed to remove any background microplastics on the materials. The procedures were conducted in the fume hood and the use of plastic materials was avoided throughout the experiment to minimize airborne and plastic contamination.

Task 1.1 Plastic Characterization. The objective is to create X-Ray photoelectric spectroscopy (XPS) and attenuated total reflection Fourier-transform infrared spectroscopy (ATIR-FTIR) databases from different types of plastics. Nine samples of different plastic materials were prepared by cutting them into smaller pieces (~1 cm^2) for analyses. The plastic materials were (1) plastic bag, (2) plastic cup, (3) Polystyrene disposable dish, (4) 100% polyester clothing, (5) 50% polyester-cotton clothing, (6) PVC tube, (7) flexible tubing, (8) nylon filter, and (9) plastic bottle. Polyethylene, polystyrene, and poly (methyl methacrylate) commercial microspheres were also included. XPS was used to examine the pre-existing metals adsorbed on the surface of the microplastics. The ATIR-FTIR recorded the IR spectra with a resolution of 4 cm^{-1}, 64 scans, and sensitivity of 1 (range: 4000-400 cm^{-1}) and gave the compound structure of each plastic material as a data base. The structure of plastic materials was analyzed using the scanning electron microscopy (SEM) and a stereo microscope up to 80x magnification to visualize the plastic structures. ZETA potential and BET analyses will also be performed on the three commercial microspheres to know the surface charge of the microplastics at different pH and to know the surface area and pore size distribution of microplastics, respectively.

Task 1.2: Detection of Microplastics. The objective is to identify microplastics and quantify the metal concentrations from freshwater samples. Task 1 identifies and characterizes microplastics

collected from freshwater systems. Samples were filtered and digested as described by Koelmans et. al (2019) to assure the quality of visual inspection and subsequent polymer identification. Water samples were weighed on a scale and shaken before filtration to maximize particle recovery from the bottle. Vacuum filtration of water samples was performed with a glass frit filter unit and 0.5 µm Advantec borosilicate glass microfiber filter in the fume hood. The filtrate water samples were recovered and transferred to clean borosilicate glasses and stored at 4°C until the quantification of heavy metal concentrations. The original sample bottle was rinsed three (3) times with ultra-pure water (18 MΩ) and passed through the vacuum filter to recover all the microplastics in the samples. Particles retained on the filter were washed into a serum bottle using 100 mL of 30% (v/v) H_2O_2 for digestion. The serum bottle was covered with aluminum foil and placed in Labnet 311DS oscillation incubator at 50°C and 80 rpm for 48 h. The first washed filter was placed in a petri dish at 4°C. After digestion, the H_2O_2 solution was vacuum filtered through a 0.5 µm borosilicate glass microfiber filter and glass frit filter unit. Lastly, the second filter was rinsed slowly with ultra-pure water (18 MΩ) to remove excess H_2O_2 solution and stored in a petri dish at 4°C for subsequent observation and identification of microplastics. A control containing only ultra-high purity water during filtering and digestion were included to quantify any interference.

Microplastics were first detected with a stereomicroscope to visualize and investigate microplastics present on the filter. This was followed by identifying microplastics with a Micro Fourier-transform infrared spectroscopy (Micro-FTIR) and X-Ray Photoelectron Spectroscopy (XPS) to determine the type of polymers and inspect the binding of metals onto the microplastics. Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDS) were used to analyze microplastic morphology and metals adsorbed on the microplastic surface, respectively. Inductively Coupled Plasma Mass Spectrometry (ICP-MS) was used to quantify the soluble metal concentrations from the filtrate water sample as mentioned above.

Task 2.1: Adsorption Kinetic Experiment. The objective is to determine the time it takes for adsorption of metals onto microplastics to reach equilibrium. Kinetic experiments were run in triplicate and used two types of heavy metals, three commercial microplastics, and ultra-pure water at pH 3 and pH 7. The series of experiments were prepared by rinsing all the glassware with 10% (v/v) HNO_3 and DI water then sonicated for 30 mins in a Cole-Parmer CPXH Series Sonicator with ultra-pure water (18 MΩ). 0.1 g of polyethylene, polystyrene, and polymethyl methacrylate commercial microspheres were weighed into each serum bottles and added 100 mL of 0.05 mM Arsenic and Uranium at pH 3 and pH 7 individually. The bottles were placed in an oscillation incubator to agitate the samples at 150 rpm for 7 days at room temperature. 1 mL of subsamples were taken at time 0, 0.25, 0.5, 1, 2, 6, 24, 50, 120, and 168 h using a syringe tip. The 0.5 mL subsamples were filtered using 0.45 µm syringe filter and then diluted 12x using 2% nitric acid, HNO_3. After 7 days, the solutions are filtered using 0.5 µm borosilicate glass microfiber filter and glass frit filter units. The collected subsamples were analyzed to quantify the soluble metal concentration using Coupled Plasma-Optical Emission Spectrometry (ICP-OES). Microplastics on the filters will be analyzed using a Micro-FTIR, XPS, and EDS to evaluate the binding of heavy metals onto microplastics. The Scanning Electron Microscopy (SEM) will be used to analyze changes in microplastic morphology.

Task 2.2 Adsorption Isotherm of Heavy Metals onto Microplastics. The objective is to assess the adsorption of four different concentrations of uranium (U) and arsenic (As) onto commercial

microplastics. All the experimental apparatus was decontaminated by rinsing with 10% (v/v) HNO_3 and DI water and was sonicated for 30 min in an ultrasonic bath with DI water before use. 0.02 g L^{-1} of polyethylene, polystyrene, and polymethyl methacrylate commercial microspheres were weighed into glass vials. Isotherms were carried out by exposing the three commercial microplastic spheres to 20mL of 0.01, 0.05, 0.1, 0.2 mM U and As for 48 h. However, exposure time could change based on the results from the kinetic experiments. Treatments were run in triplicates in an orbital shaker at 150 rpm at room temperature (25°C). Standards and blanks were also included for control. After the adsorption process, the sample solutions were vacuum filtered through a 0.5 µm borosilicate glass microfiber filter and glass frit filter unit. The filtrate water samples were transferred in a centrifuge tube and the filters were placed in a petri dish and stored at 4°C. The size and morphology of the microplastic spheres were confirmed with SEM and EDS, respectively. Metal adsorption was determined by quantifying the soluble metal concentration with Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES). Micro-FTIR and XPS were used to evaluate the binding of heavy metals onto microplastics.

Results

Microplastic Characterization

Nine samples of different plastic materials and three (3) commercial microplastics were analyzed using the ATIR-FTIR and XPS. These analyses served as databases of different types of plastics as shown in **Figures 2 to 5** and **Tables 1 to 4**. In addition, SEM and EDS analyses were performed on the three commercial microplastics to investigate the morphology and surface of the microplastics (**Figures 6 to 11**). See appendix section (**Figures 22A to 32A**) for the XPS and SEM/EDS data of other plastic materials. These collected inputs will aid the identification of microplastics in freshwater systems in NM and will give information on the structure and surface of the commercial microplastics used for Task 2.1 and 2.2 experiments.

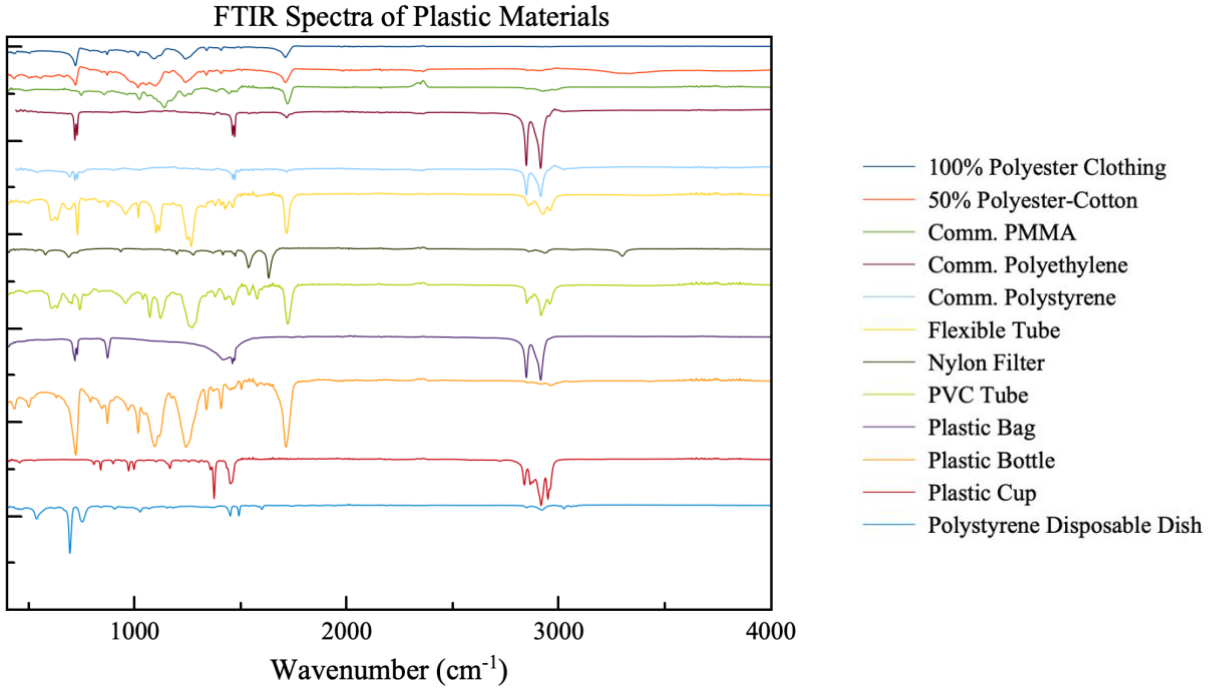

Figure 2: FTIR spectra of different plastic materials with a resolution of 4 cm^{-1}, 64 scans, and sensitivity of 1 (range: 4000-400 cm^{-1}).

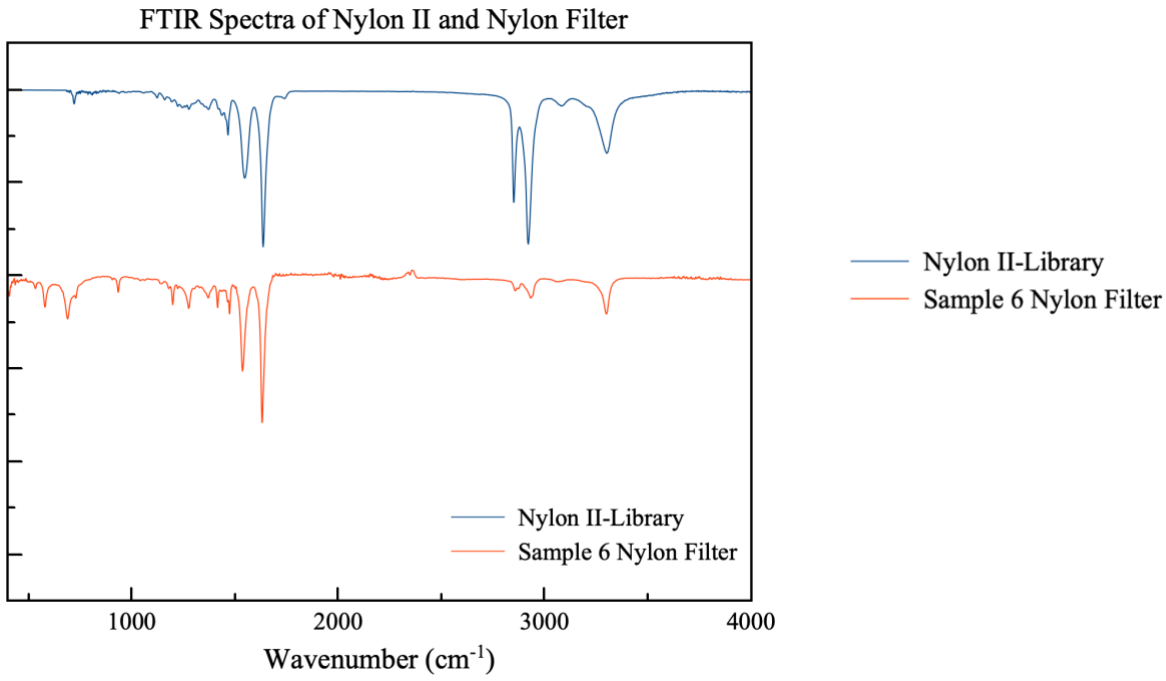

Figure 3: FTIR spectra of nylon II from ATIR-FTIR library and sample 6 nylon filter.

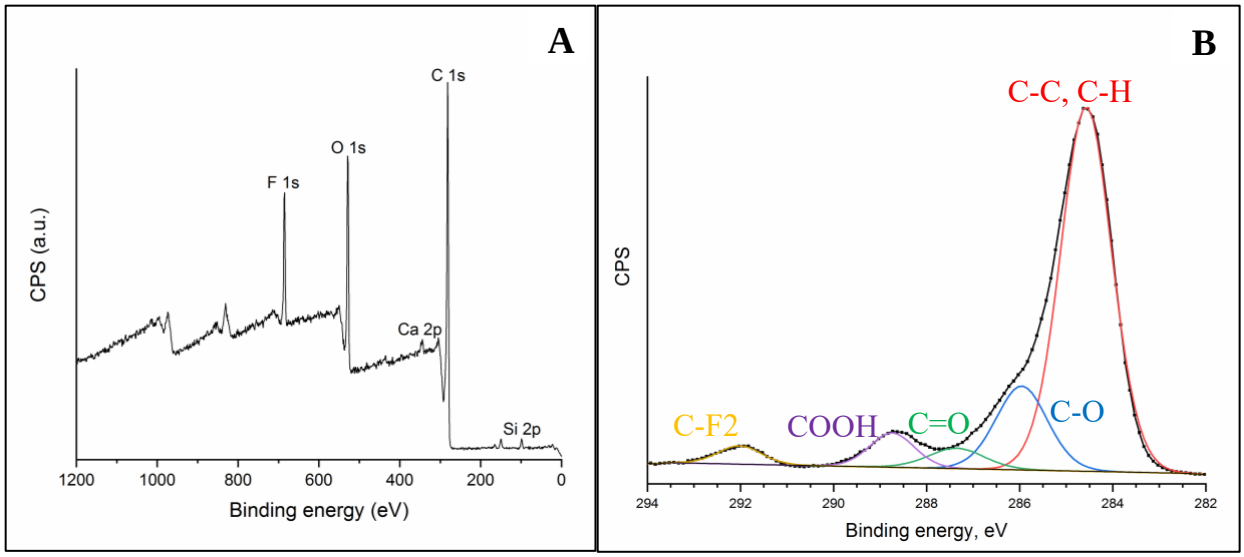

Figure 4: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of 100% polyester clothing.

Table 1: Area percentages of each element from 100% polyester clothing survey scan.

C 1s %	Ca 2p %	F 1s %	O 1s %	Si 2p %
78.11 ± 2.41	0.67 ± 0.25	5.27 ± 1.92	14.26 ± 0.41	1.68 ± 0.29

Table 2: Area percentages of C 1s functional groups from 100% polyester clothing.

C-C, C-H %	C-F2 %	C-O %	C=O %	COOH%
69.89 ± 1.49	2.76 ± 1.37	17.00 ± 0.65	4.24 ± 0.15	6.11 ± 0.56

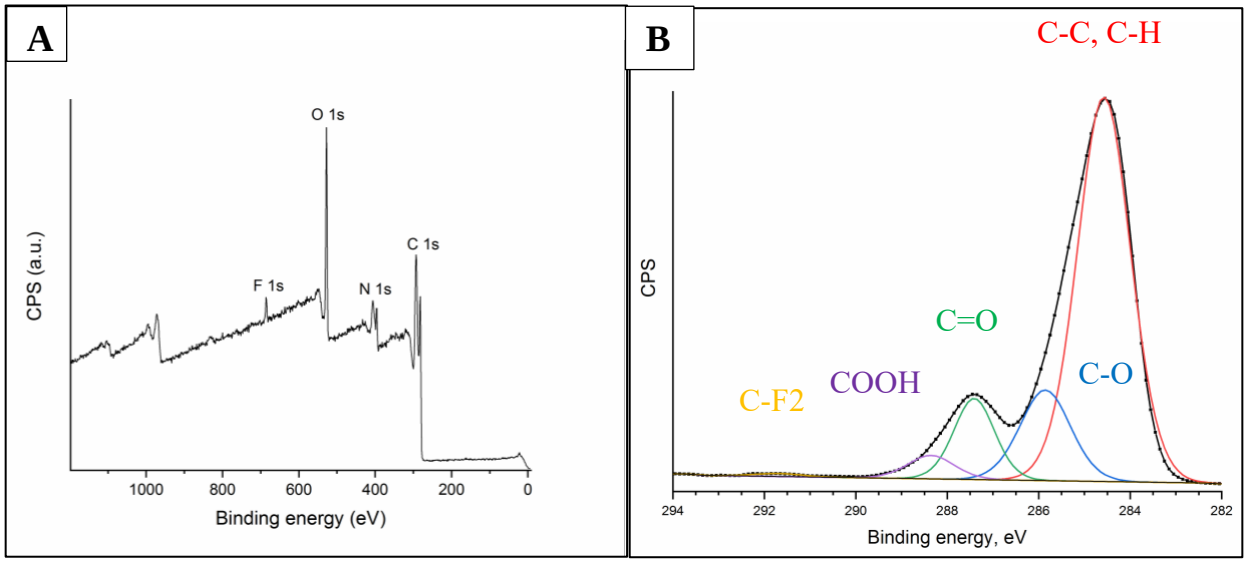

Figure 5: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of nylon filter.

Table 3: Area percentages of each element from nylon filter clothing survey scan.

C 1s %	F 1s %	N 1s %	O 1s %
77.17 ± 1.77	0.88 ± 0.39	10.82 ± 0.93	11.13 ± 1.17

Table 4: Area percentages of C 1s functional groups from nylon filter.

C-C, C-H %	C-F2 %	C-O %	C=O %	COOH%
69.31 ± 0.34	0.51 ± 0.02	14.51 ± 1.43	11.72 ± 0.73	3.95 ± 0.34

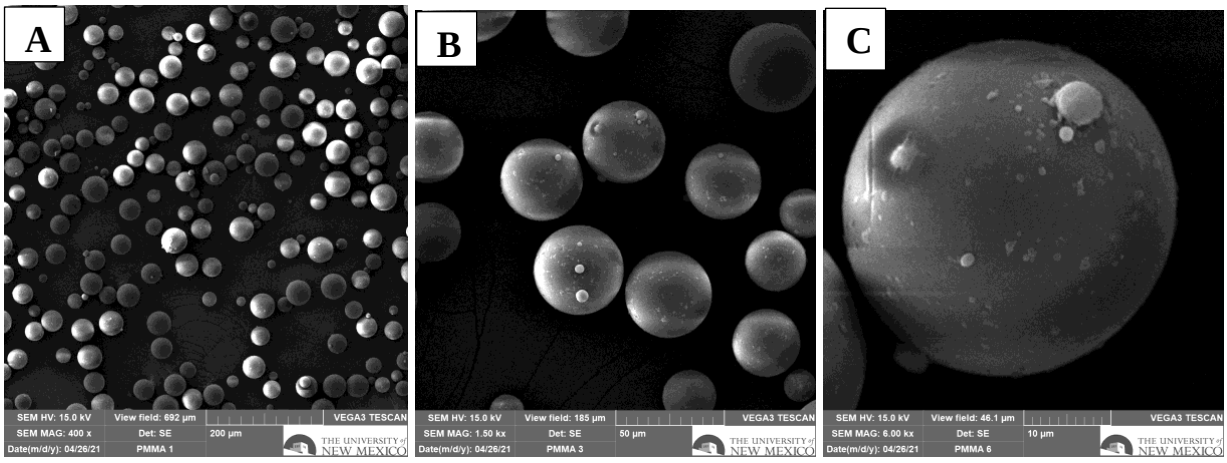

Figure 6: SEM images of poly (methyl methacrylate) commercial microspheres at different magnifications.

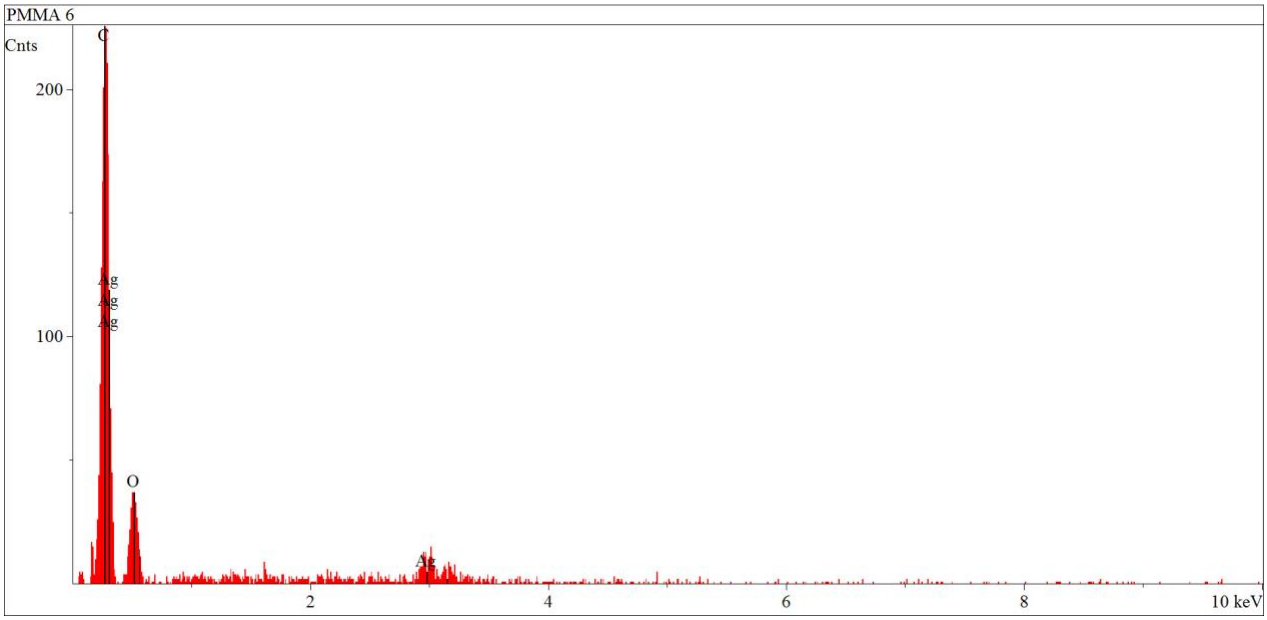

Figure 7: EDS spectrum of poly (methyl methacrylate) commercial microspheres.

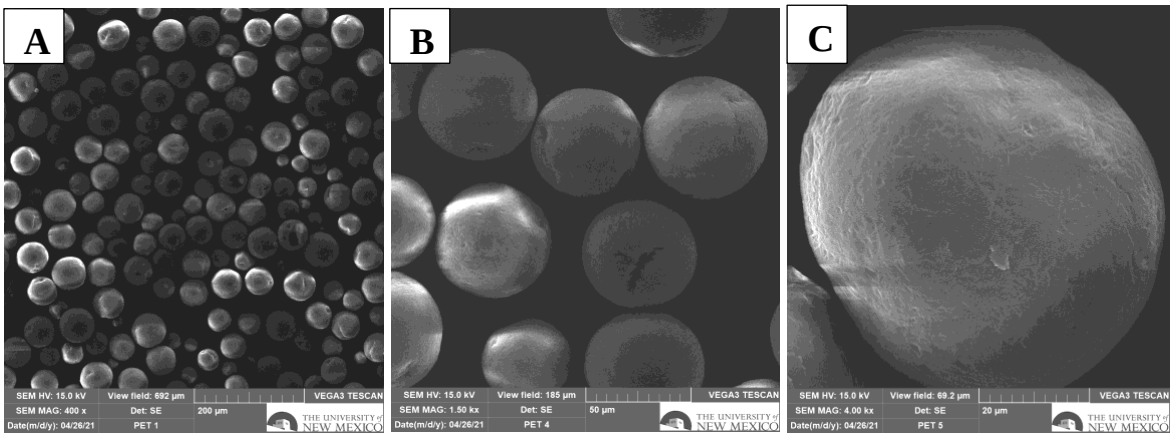

Figure 8: SEM images of polyethylene commercial microspheres at different magnifications.

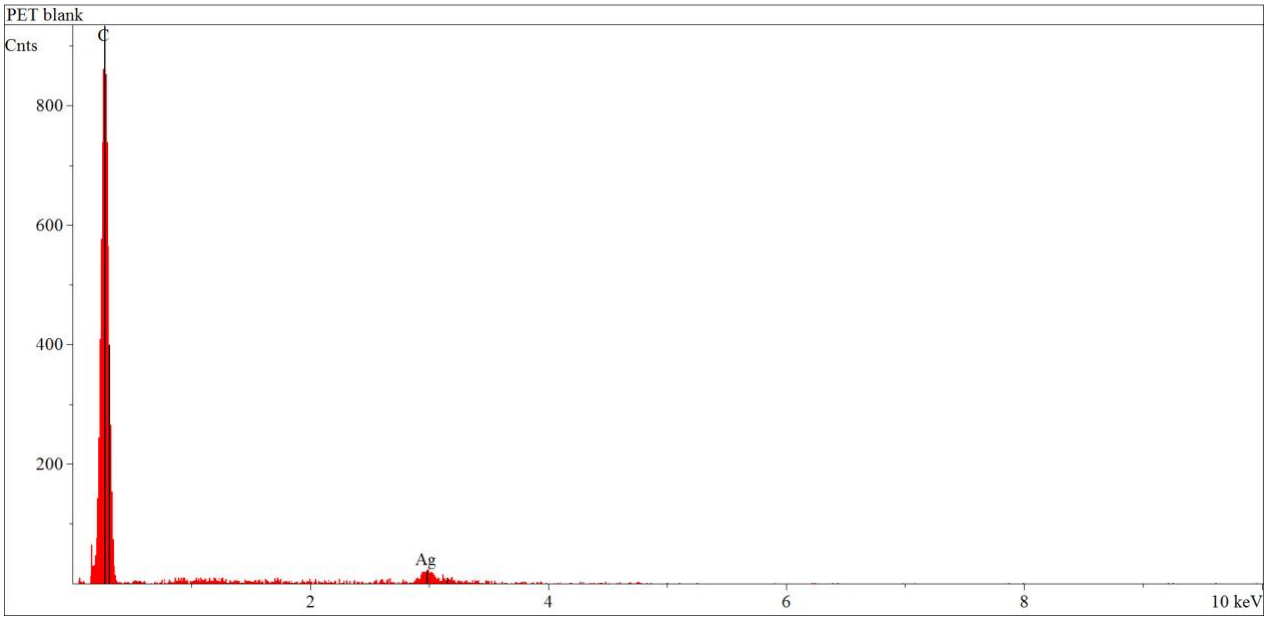

Figure 9: EDS spectrum of polyethylene commercial microspheres.

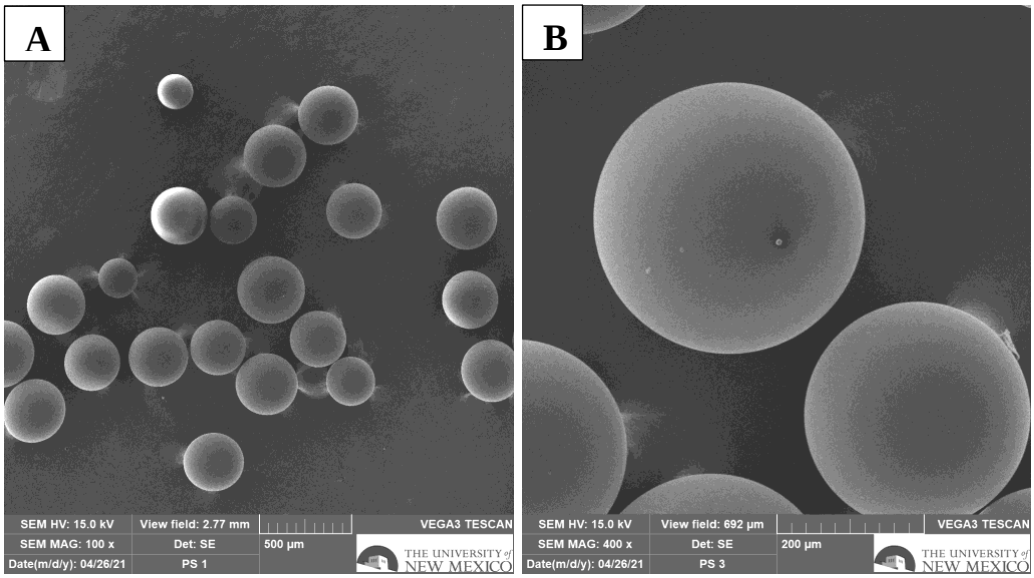

Figure 10: SEM images of polystyrene commercial microspheres at different magnifications.

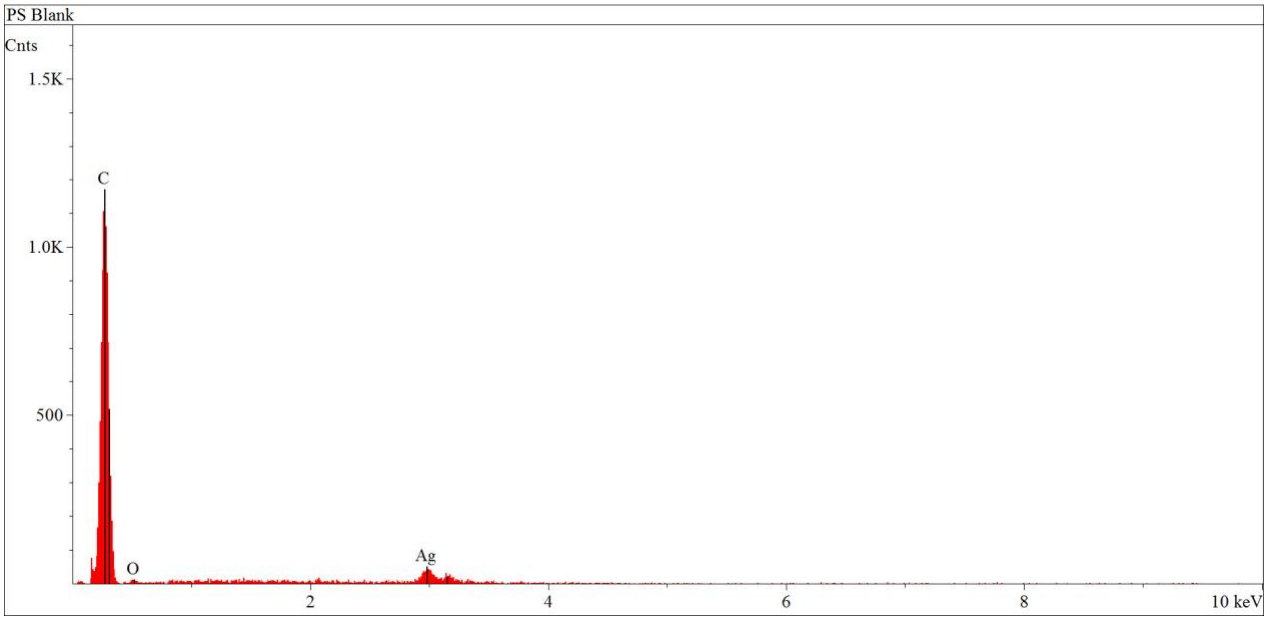

Figure 11: EDS spectrum of polystyrene commercial microspheres.

Detection of Microplastics in NM Freshwater System

Table 5 shows the As and U concentrations of freshwater systems in NM (e.g., Tingley Beach, Rio Grande, Paguate River, and freshwater reservoirs in Laguna Pueblo). The data indicated concentrations of As in the Tingley Beach samples above the USEPA maximum contaminant levels (MCL) of 10 μg/L for drinking water.[6] Furthermore, Laguna samples 4-6 (Table 5) show relatively high U concentration and coincide with the findings of Blake et. al.[3] The previous study also showed the concentration of U exceeding the USEPA maximum contaminant levels (MCL) of 30 μg/L for drinking water.[3,6] The data found in this section indicates the significant U and As concentrations present in freshwater systems in New Mexico.

Table 5: Arsenic (As) and uranium (U) concentrations (μg/L) from different sampling locations in New Mexico freshwater systems.

Sample Name	Arsenic (μg/L)	Uranium (μg/L)
Tingley Beach 1	10.75 $\pm$ 0.33	2.31 $\pm$ 0.23
Tingley Beach 2	11.40 $\pm$ 0.24	2.35 $\pm$ 0.39
Tingley Beach 3	11.26 $\pm$ 0.20	2.16 $\pm$ 0.12
Rio Grande 2 - State Road NM	3.10 $\pm$ 0.08	0.97 $\pm$ 0.09
Rio Grande 3 - State Road, NM	1.59 $\pm$ 0.12	1.39 $\pm$ 0.07
Rio Grande 4 – Espanola, NM	2.03 $\pm$ 0.20	2.18 $\pm$ 0.14
Rio Grande 4 – Albuquerque	2.32 $\pm$ 0.09	1.07 $\pm$ 0.34
Rio Grande 5 – Albuquerque	3.01 $\pm$ 0.04	1.43 $\pm$ 0.23
Rio Grande 6 – Albuquerque	3.05 $\pm$ 0.48	1.34 $\pm$ 0.28
Laguna Sample 1	2.27 $\pm$ 0.06	0.65 $\pm$ 0.08
Laguna Sample 2	1.45 $\pm$ 0.07	0.45 $\pm$ 0.11
Laguna Sample 3	0.81 $\pm$ 0.09	1.85 $\pm$ 0.21
Laguna Sample 4	0.66 $\pm$ 0.06	36.53 $\pm$ 3.02
Laguna Sample 5	1.43 $\pm$ 0.13	28.27 $\pm$ 1.61

Laguna Sample 6	1.30 ± 0.03	29.65 ± 2.03
Field Control	0.43 ± 0.02	0.13 ± 0.06
Procedure Control	0.42 ± 0.05	0.13 ± 0.09

Adsorption Kinetic Experiment

Adsorption kinetic experiments were performed on the three commercial microplastics (PMMA, PE, and PS) and arsenic (As) at pH 3 and pH 7 for 7 days. The data shown in **Figures 12 to 17** indicate the low adsorption capacity of As and the commercial microplastics. This could be due to the insufficient charge differential between As and the microplastics where adsorption depends on weak forces such as Van der Waal forces. The next batch of experiments will be uranium (U) and the three microplastics. We hypothesized that the negative charges of polystyrene and polyethylene microplastics will attract U positively charged at pH 3 and pH 7.[7,8] This hypothesis is supported by Tourinho et. al[9], their findings stated that the negative net charges on polystyrene and polyethylene surfaces are likely to attract positively charged species.[9] The data collected from this section is significant to understand the synergy of these two pollutants which could potentially increase toxic effects on human health and the environment.

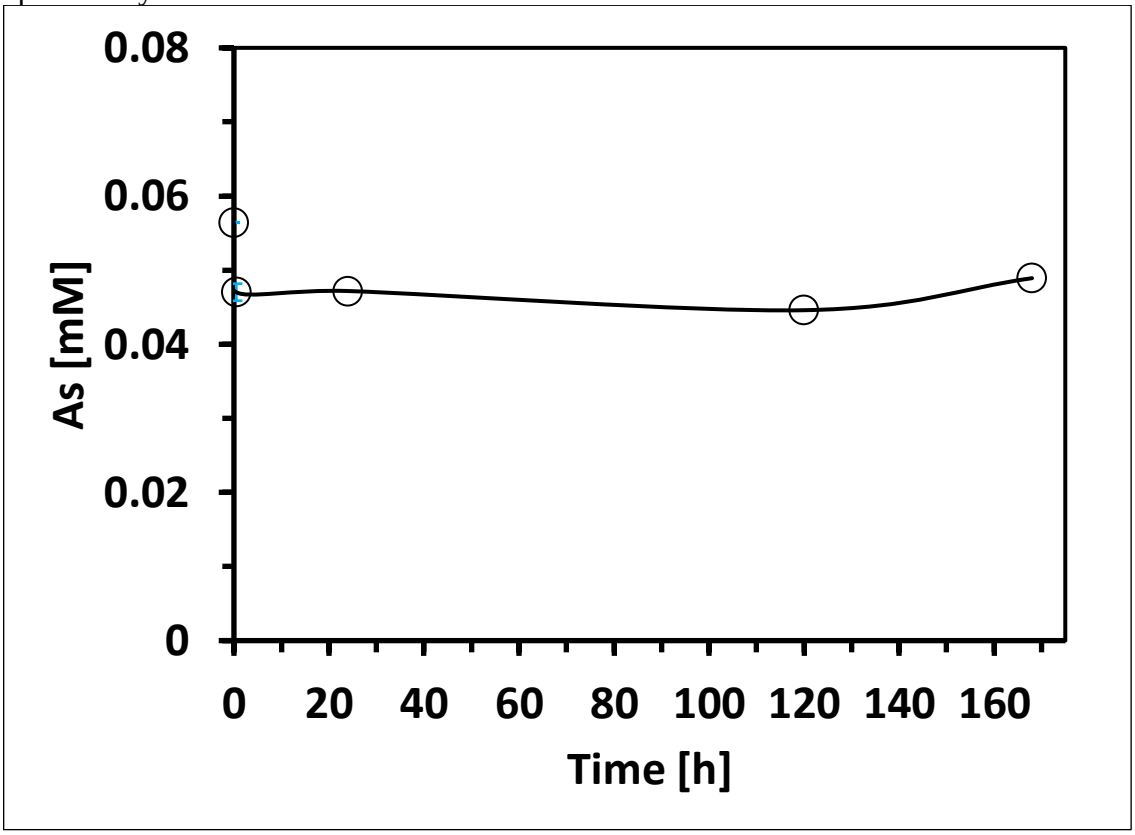

Figure 12: Adsorption kinetics of As onto PMMA at pH 3 after 7 days. The results are from a preliminary experiment. Error bars indicate standard deviation obtained from duplicates.

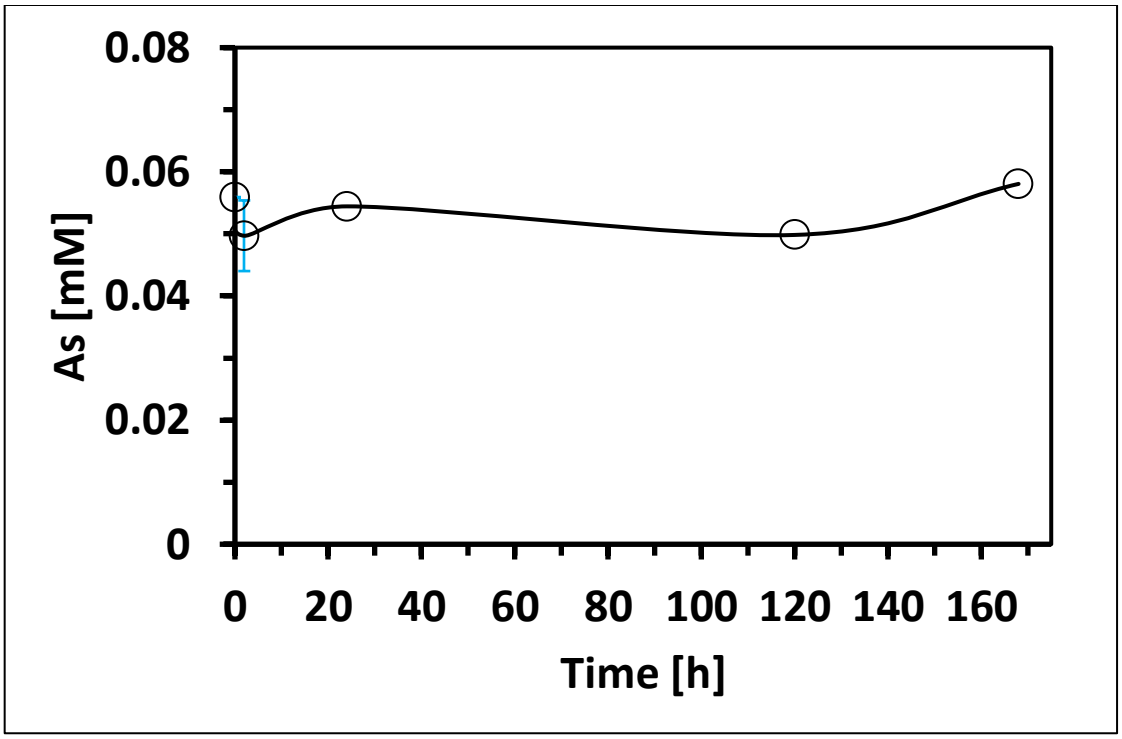

Figure 13: Adsorption kinetics of As onto PMMA at pH 7 after 7 days. The results are from a preliminary experiment. Error bars indicate standard deviation obtained from duplicates.

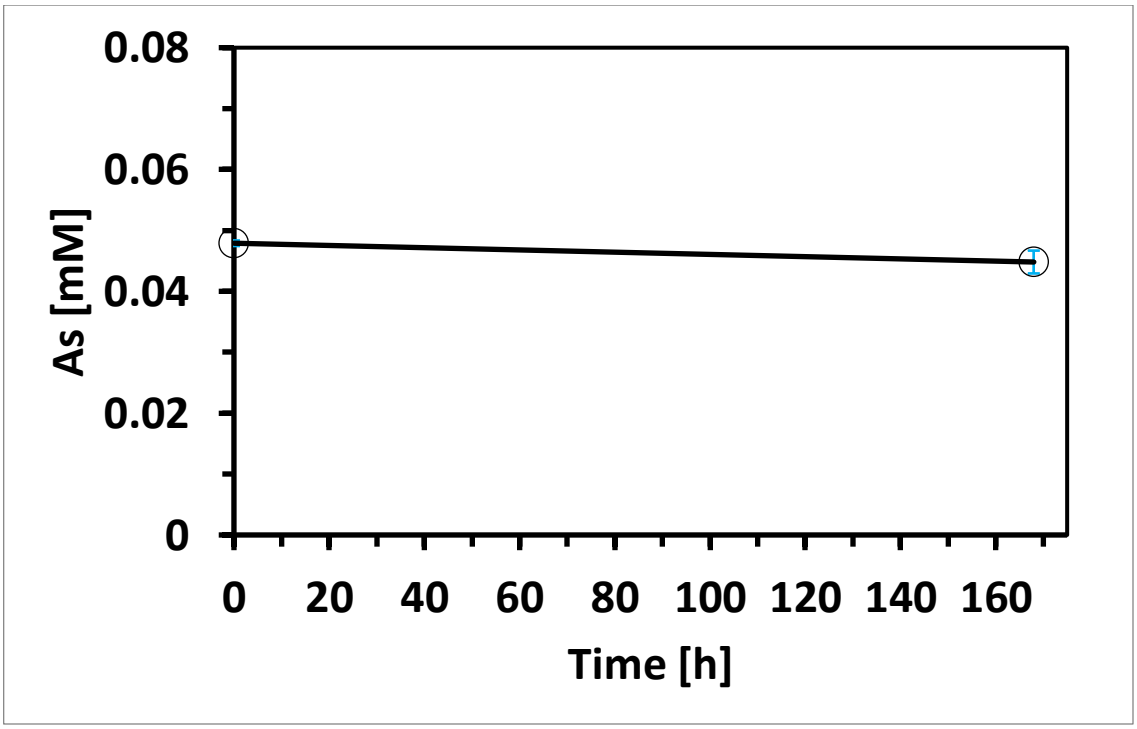

Figure 14: Adsorption kinetics of As onto PS at pH 3 after 7 days. Error bars indicate standard deviation obtained from triplicate.

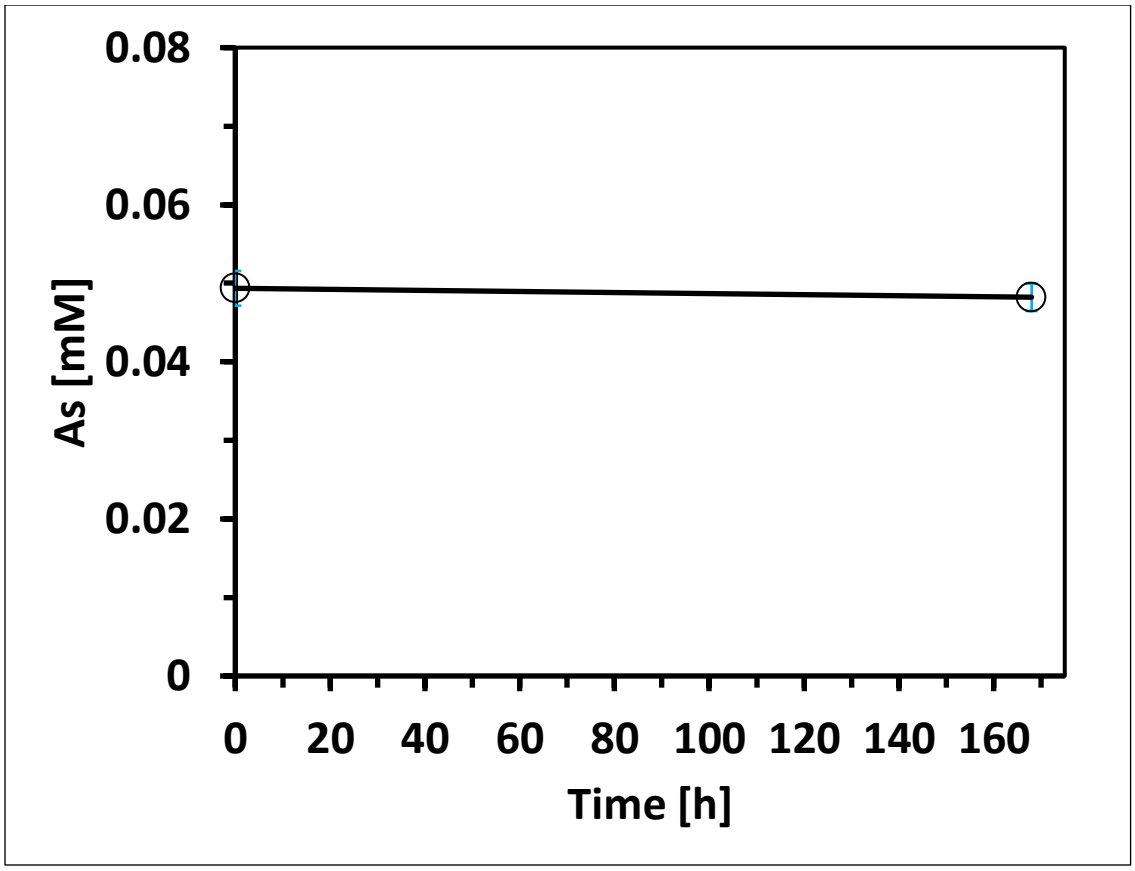

Figure 15: Adsorption kinetics of As onto PS at pH 7 after 7 days. Error bars indicate standard deviation obtained from triplicate.

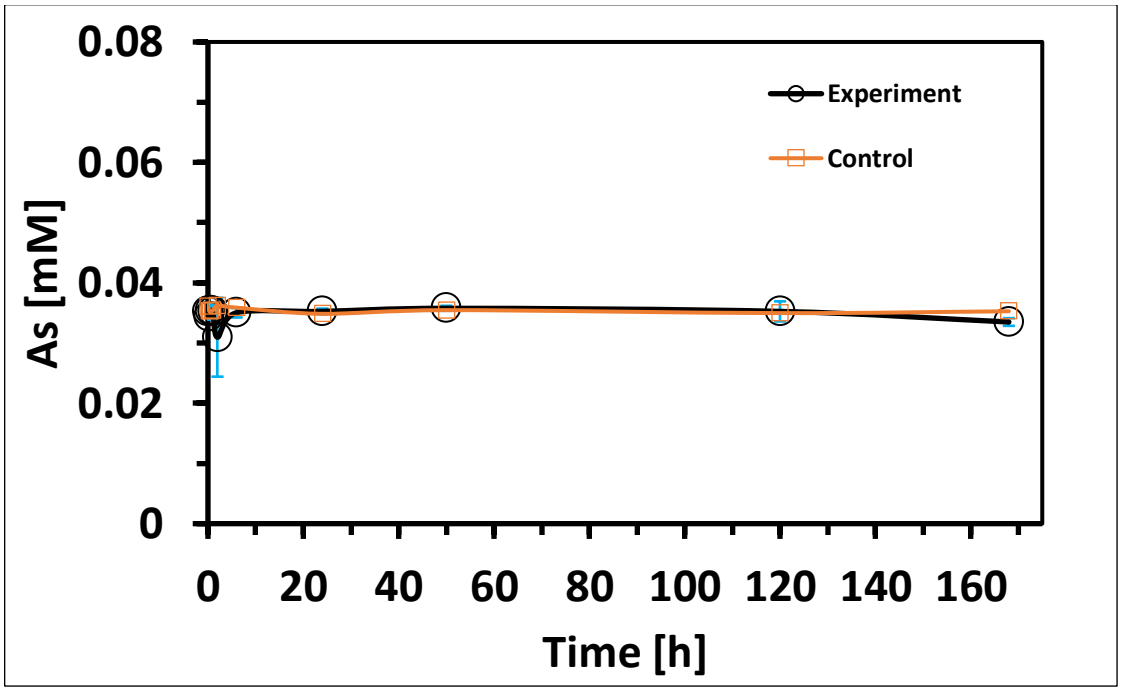

Figure 16: Adsorption kinetics of As onto PE at pH 3 after 7 days. Error bars indicate standard deviation obtained from triplicate.

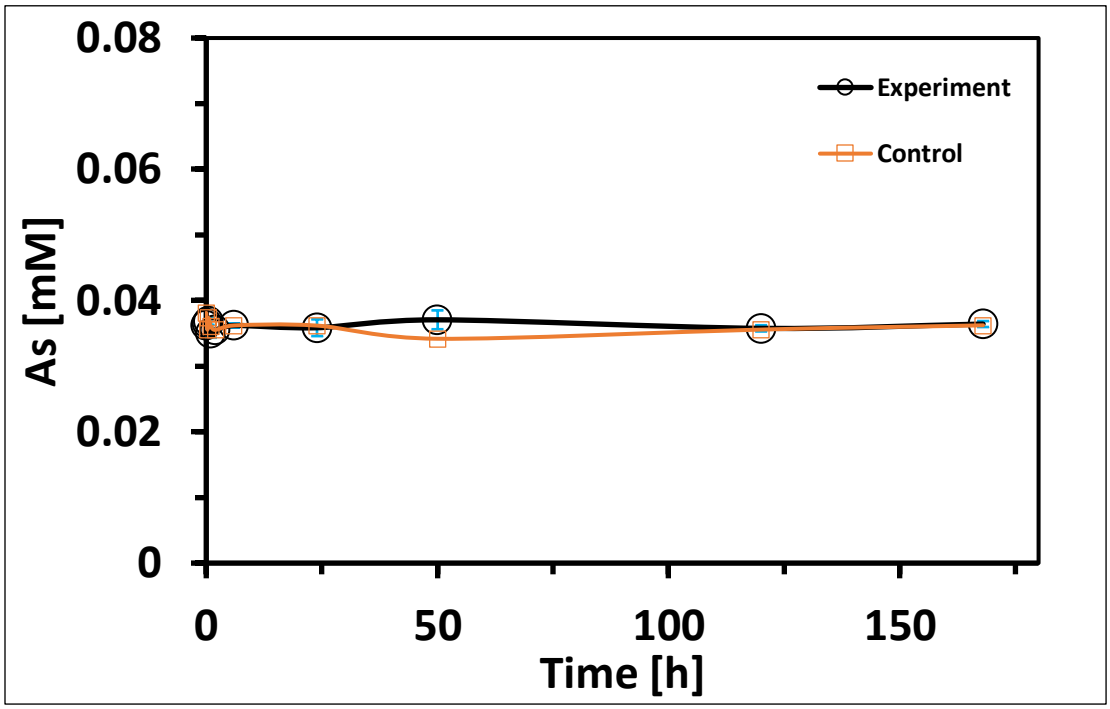

Figure 17: Adsorption kinetics of As onto PE at pH 7 after 7 days. Error bars indicate standard deviation obtained from triplicate.

Adsorption Isotherm of Heavy Metals onto Microplastics

Figures 18 to 21 show the morphology and surface of the PMMA commercial microplastics after exposing it to 0.2mM As concentration at pH 3 and pH 7 for 48 hr. The data shown here confirmed that adsorption of As onto the microplastics surface did not take place after 48 hr.

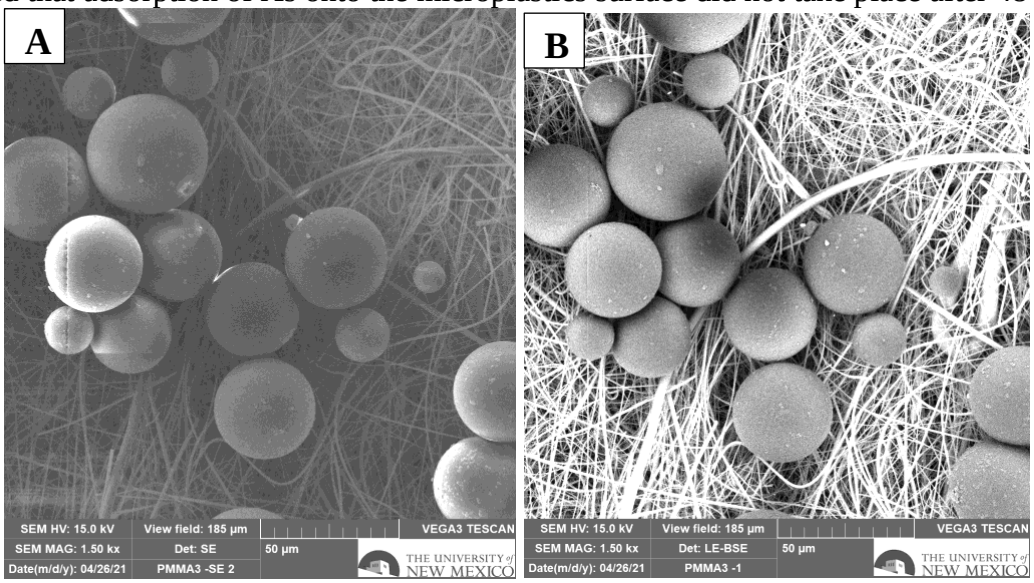

Figure 18: SEM and backscatter (BSE) images of PMMA on glass microfiber filter after 48 hr exposure on 0.2 mM As at pH 3.

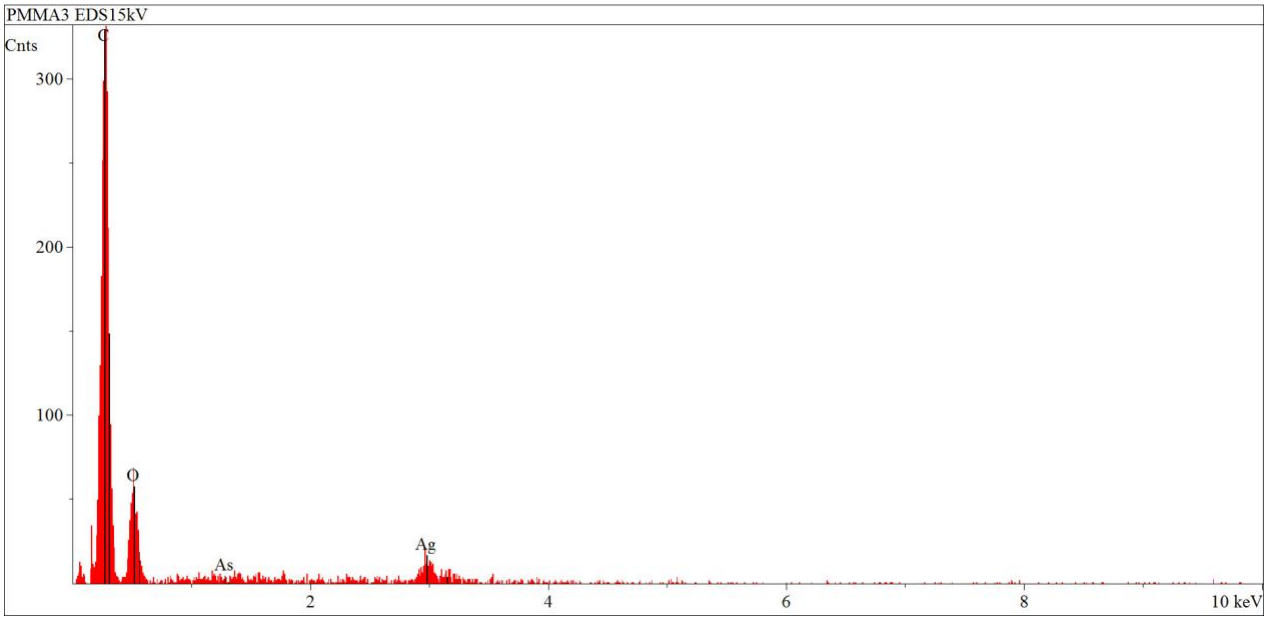

Figure 19: EDS spectrum of PMMA after 48 hr exposure on 0.2 mM As at pH 3.

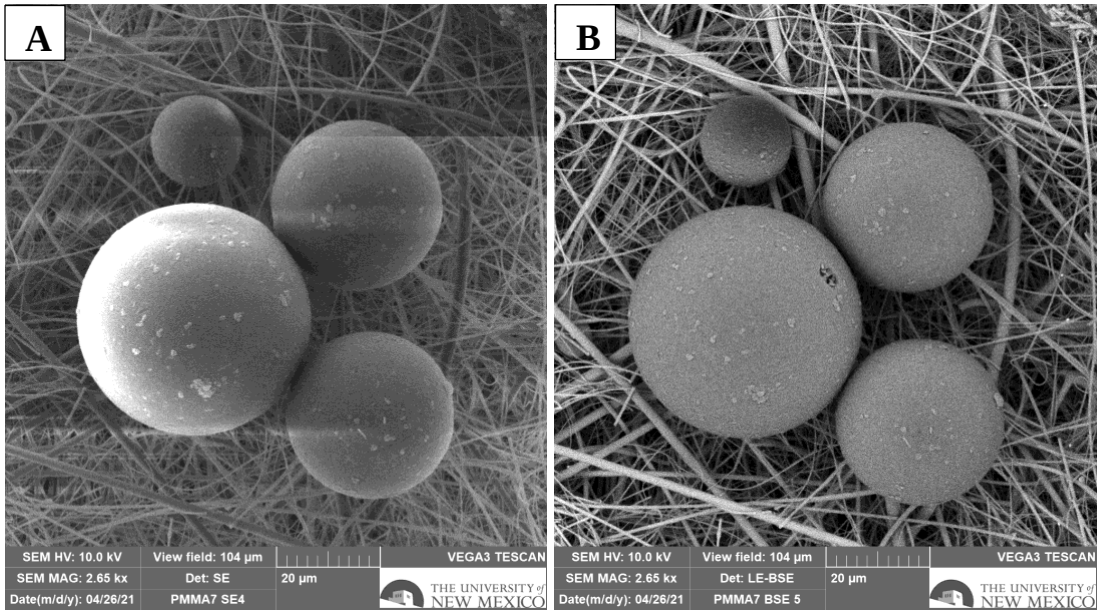

Figure 20: SEM and backscatter (BSE) images of PMMA on glass microfiber filter after 48 hr exposure on 0.2 mM As at pH 7.

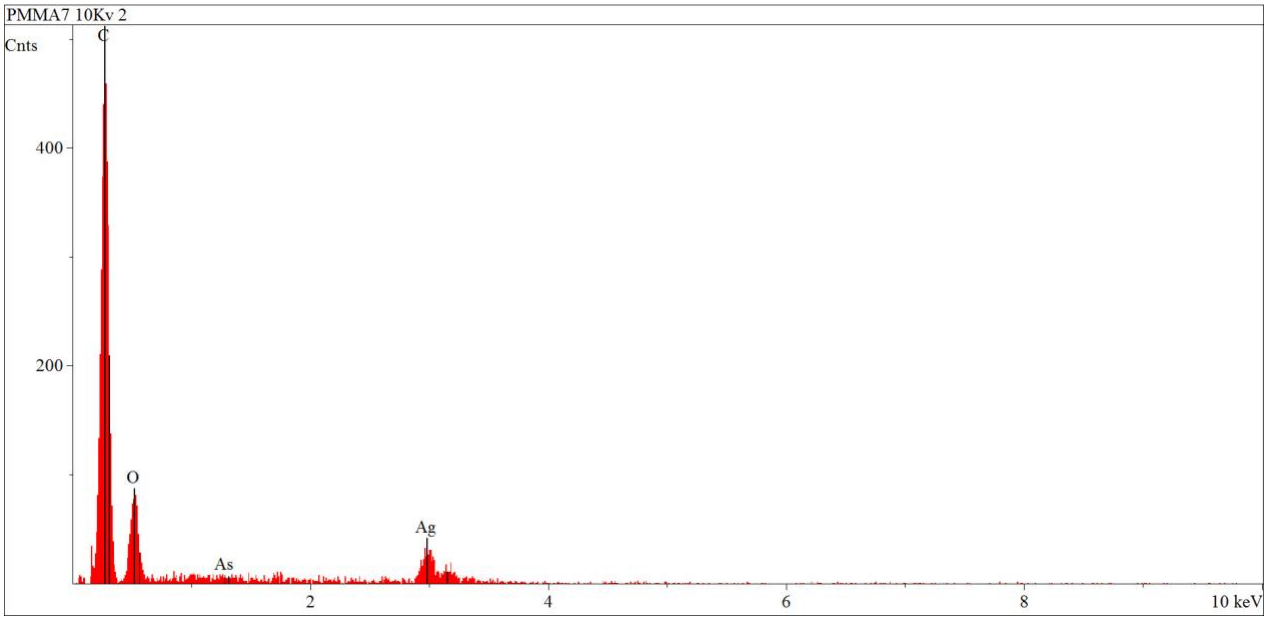

Figure 21: EDS spectrum of PMMA after 48 hr exposure on 0.2 mM As at pH 3.

Who will benefit from this research results?

The findings of this research study will inform the New Mexico Water Resources Research Institute (NMWRRI) and other researchers of potential adsorption of heavy metals onto microplastics in freshwater systems where it could potentially open new research areas. The public especially the New Mexico residents and the Laguna Pueblo community will also benefit from this research because it will give awareness of their possible interaction with microplastics and high concentrations of heavy metals in their local freshwater system. The accumulation of microplastics and the elevated heavy metal concentrations in freshwater systems in the state of New Mexico near abandoned mine sites could enhance the toxic effects on humans and the environment. This research will serve as a basis for future studies to understand the synergies of these contaminants and their toxic environmental effects.

How have you spent your grant funds?

Transaction Description	Budget	Actual Expenditures
Salaries	3,500	3,501.
Other Staff Benefits	635	275.51
Chemicals	0	310.72
Supplies	340	1,060.54
In State Travel	505	.00
FTIR Analyses	700	.00
SEM/EDS Analyses	700	.00
ICP-OES Analyses	700	1,912.50
Technical Services Gen (Total)	2,100	1,912.50
Total	7,080.00	7060.27
Total Budget Remaining		19.73

NMWRRI grant's funds were entirely spent for this research project. The in-state travel fund was used for supplies and chemicals and technical services fund was expended for ICP-OES analysis.

Presentations made related to the project

- The 65th Annual NMWRRI Conference (Poster Presentation)
- GE^3 Research Group Meeting (January 21st, 2021)
- The upcoming 1st Annual Mini Conference of Center for Water & the Environment (CWE)

Publications or Reports

A thesis project for my master's degree in Environmental Engineering in the Civil Engineering department at the University of New Mexico, and a scientific manuscript.

List of other students or faculty members who have assisted you with your project

Advisor: Jorge Gonzalez-Estrella, Assistant Professor at Oklahoma State University
Co-Advisor: Dr. Kerry Howe, Professor and the Director of Center for Water and the Environment at the University of New Mexico
Angelica Benavidez, Research Assistant Professor at the University of New Mexico
Carmen Velasco, Postdoctoral Research Associate, Arizona State University
Katelin Fisher, Environmental Laboratory Manager at the University of New Mexico
Jose Cerrato, Associate Professor at the University of New Mexico
Dr. Abdul Mehdi S. Ali, Senior Research Scientist II at the University of New Mexico
Isabel Meza, PhD Candidate at the University of New Mexico
Roxanne Awais, Graduate Research Assistant at the University of New Mexico
Michael N. Spilde, Manager, Microprobe/SEM Laboratories at the University of New Mexico

Special recognition awards or notable achievements

New Mexico Water Resources Research Institute (NMWRRI) eNews in November 2020

Degree of completion and future career plans

I am planning to complete my master's degree in Environmental Engineering at the University of New Mexico by December 2021. After my degree completion, I plan to get a job in any water industries in New Mexico. I am interested in applying to Intel in a water-related field job, any wastewater treatment plants, or the Albuquerque Water Utility Authority company. I have a wide variety of options, but I want to focus on working on water and environment-related jobs once I get my degree.

References

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Appendix

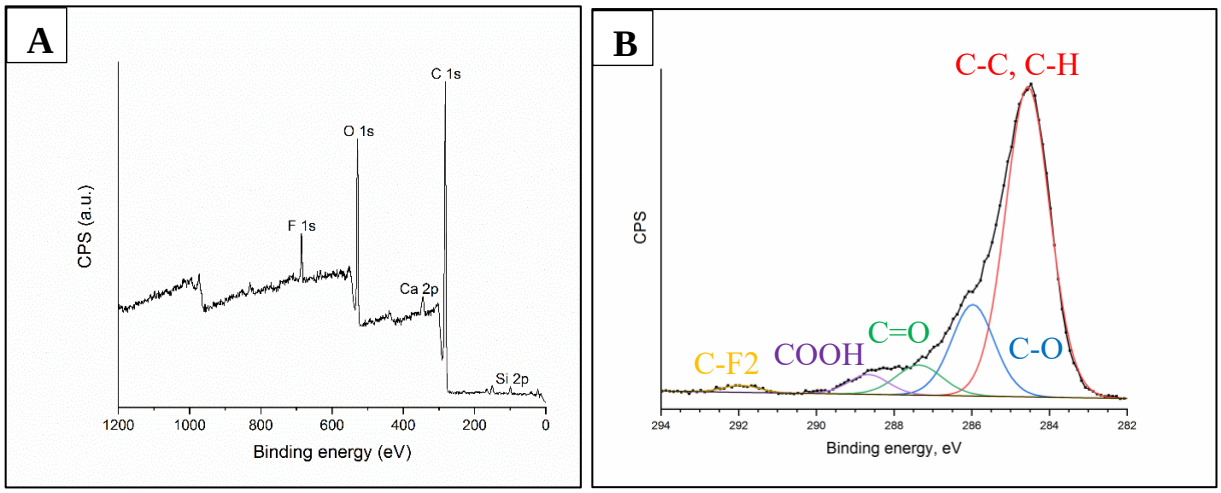

Figure 22A: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of 50% polyester and 50% cotton clothing.

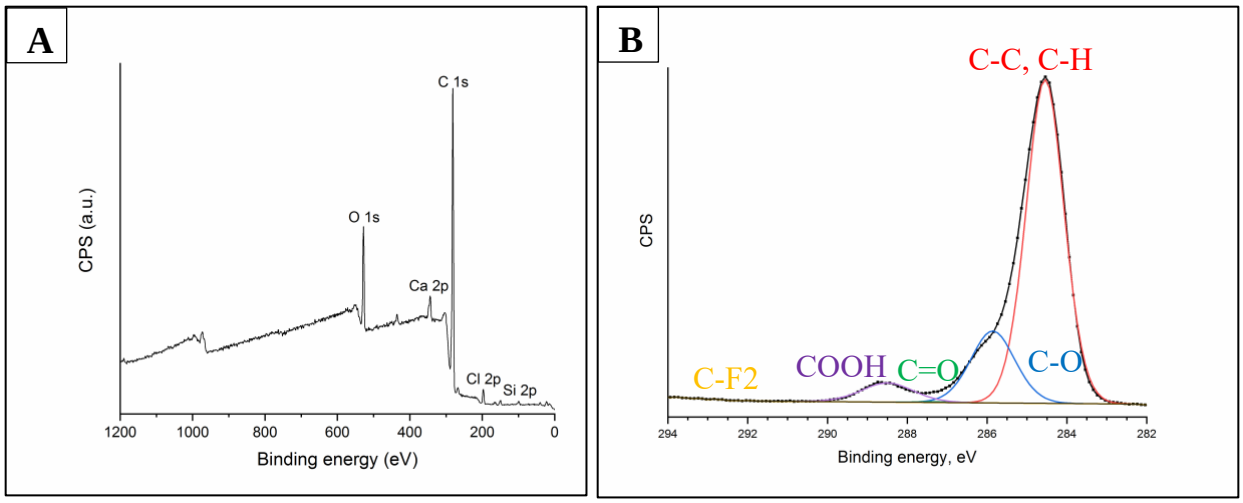

Figure 23A: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of flexible tube.

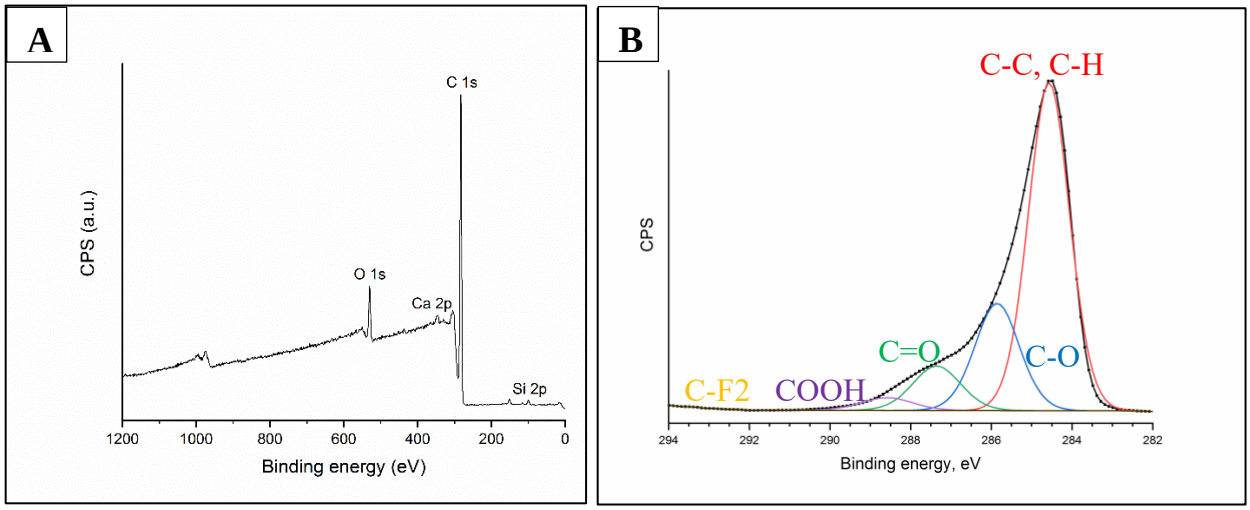

Figure 24A: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of plastic bag.

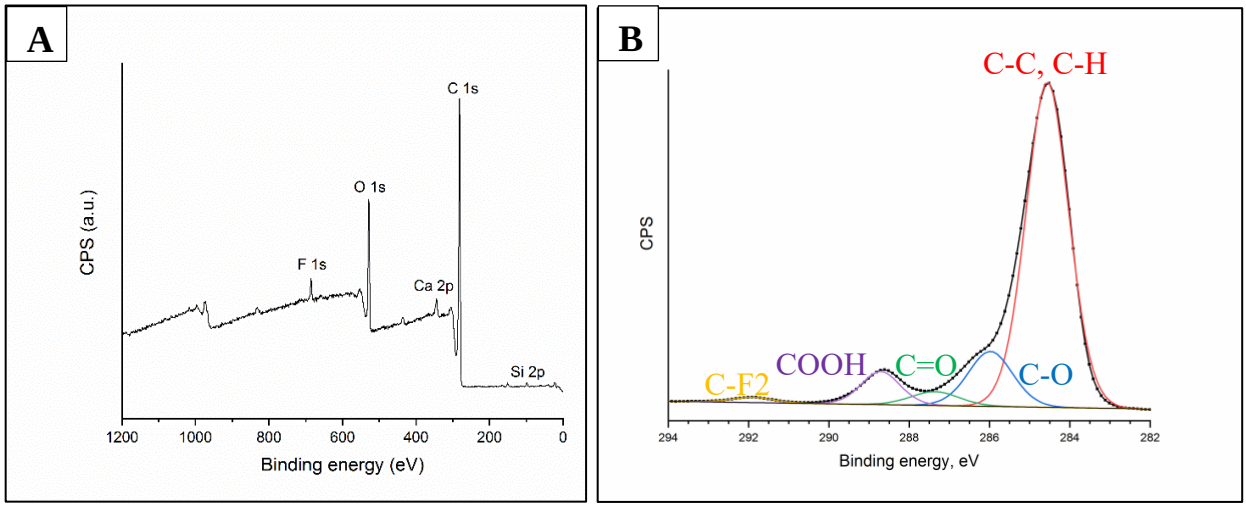

Figure 25A: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of plastic bottle.

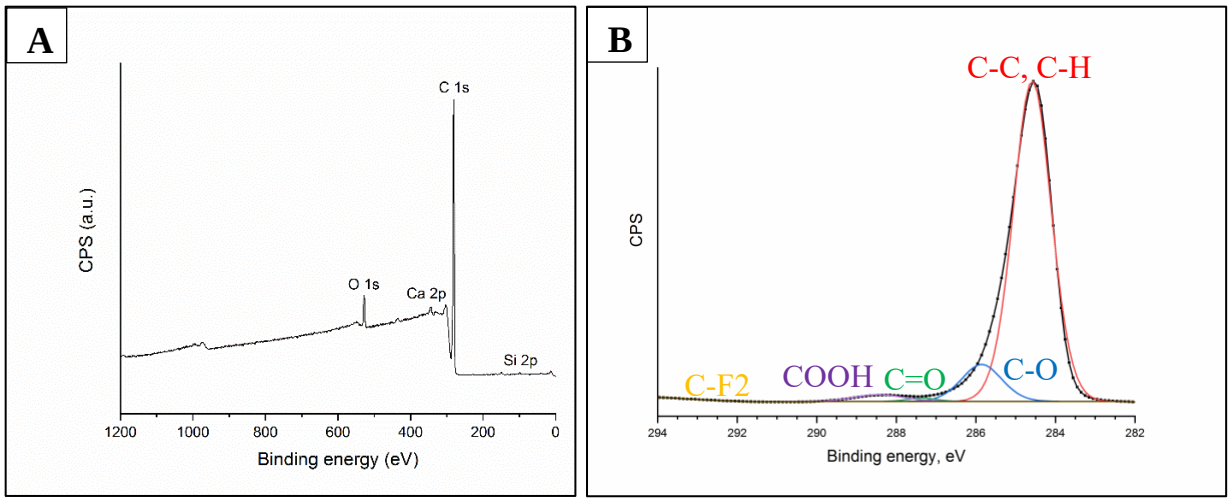

Figure 26A: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of plastic cup.

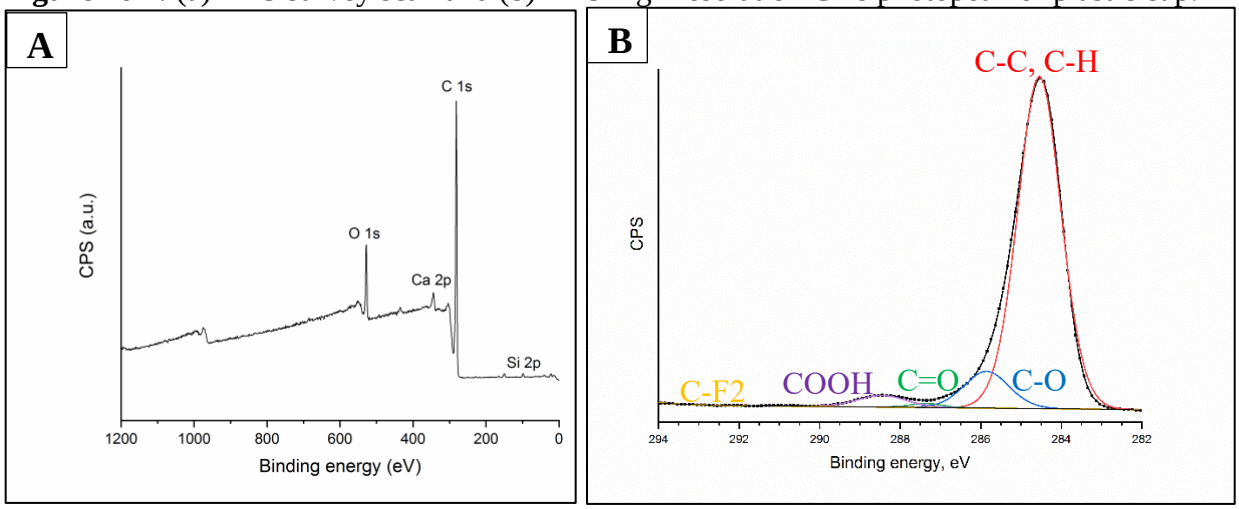

Figure 27A: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of polystyrene dish.

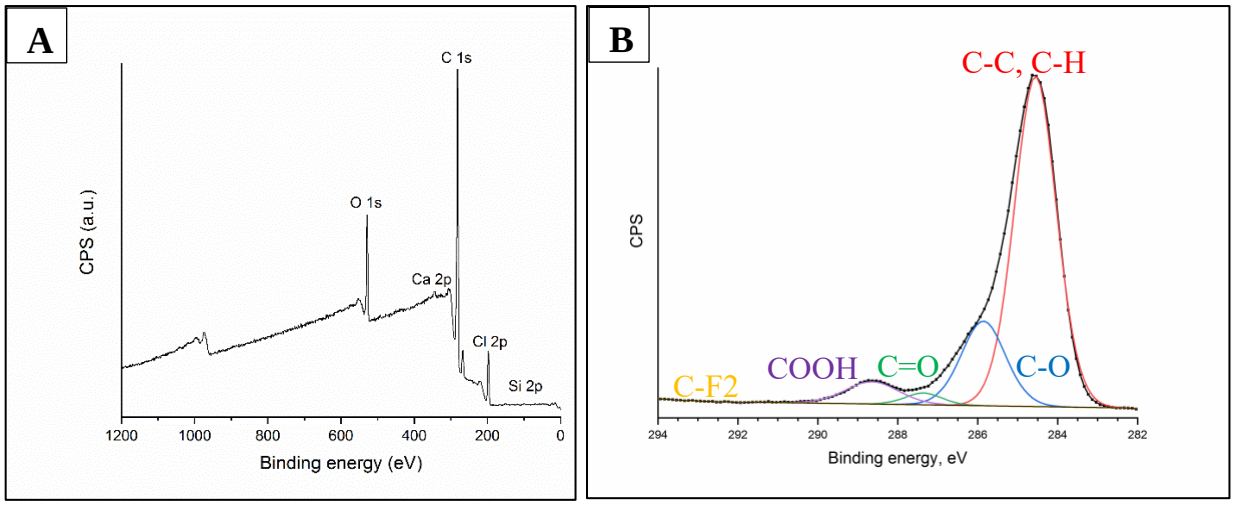

Figure 28A: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of PVC tube.

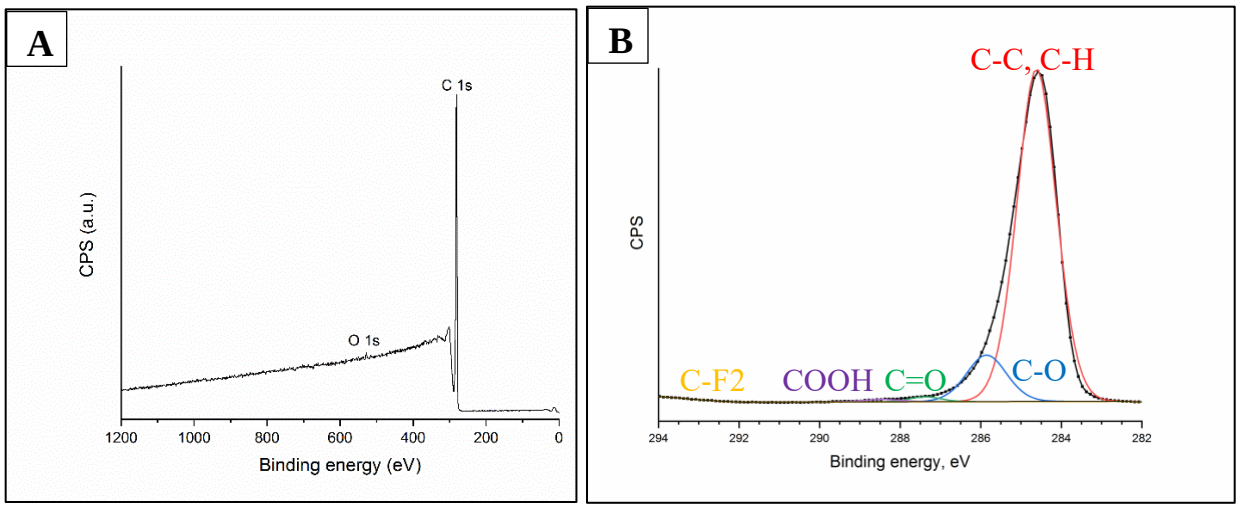

Figure 29A: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of commercial polyethylene microspheres.

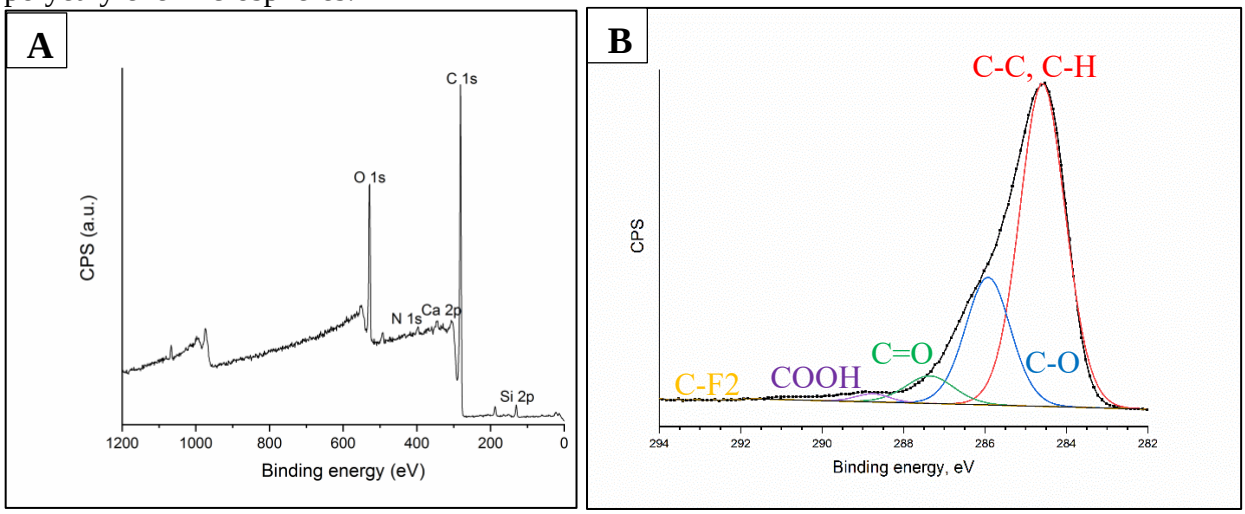

Figure 30A: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of commercial polystyrene microspheres.

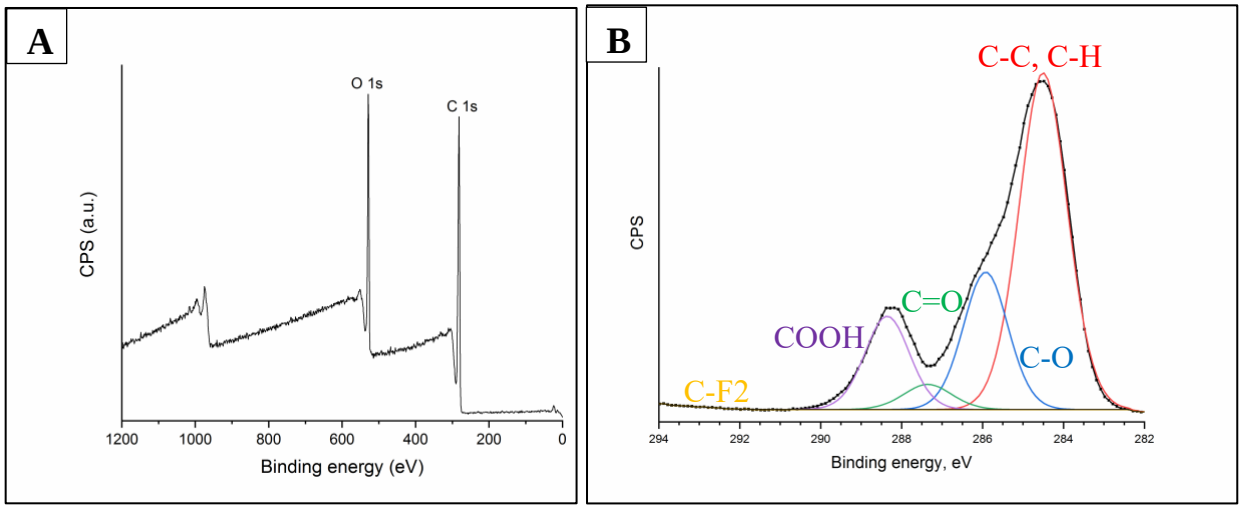

Figure 31A: (a) XPS survey scan and (b) XPS high resolution C 1s photopeak of commercial poly(methyl methacrylate) microspheres.

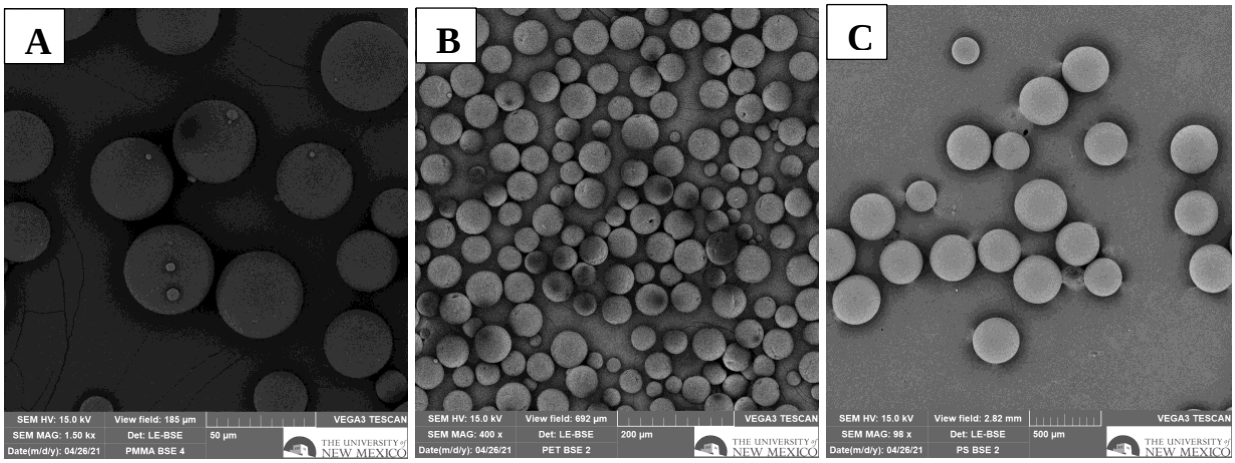

Figure 32A: BSE images of (a) PMMA, (b) PE, and (c) PS commercial microspheres at different magnifications.

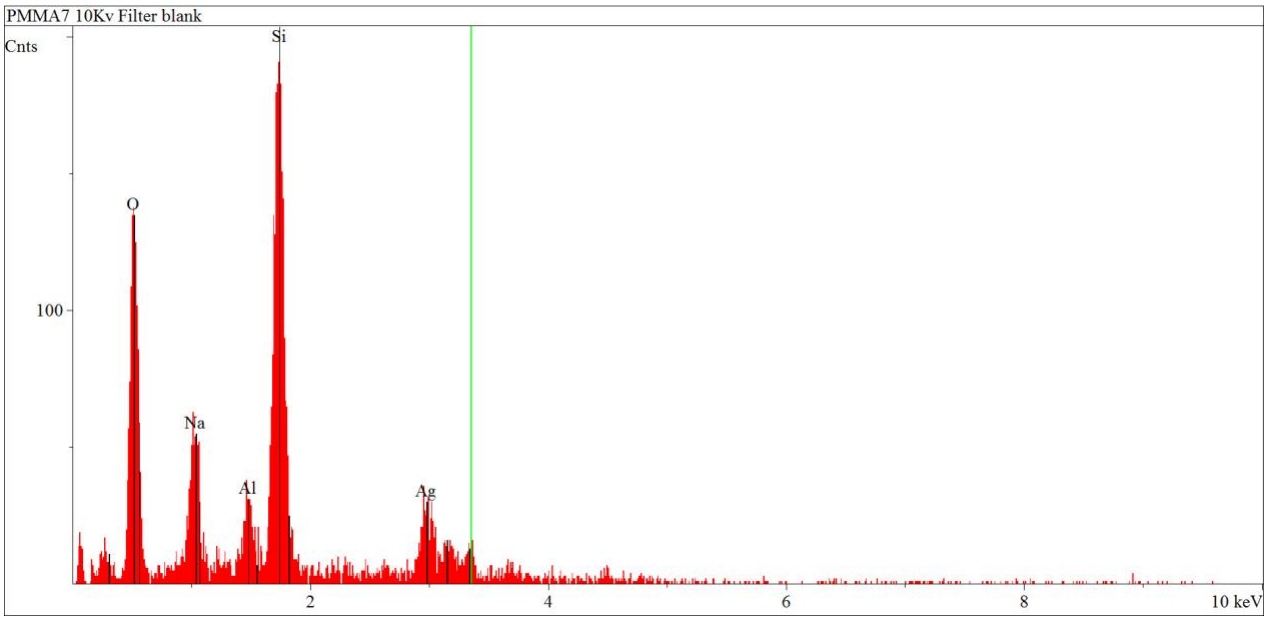

Figure 33A: EDS spectrum of 0.5 µm Advantec borosilicate glass microfiber filter for control.