

Americium Sorption by Microplastics in Aqueous Solutions

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Abstract: The interaction of americium by polyamide (PN6) and polyethylene (PE) microplastics (MPs) has been investigated in de-ionized and seawater samples traced with the Am-241 isotope. The effect of the pH and composition of the aqueous solution (seawater) on the sorption efficiency (K_d) have been studied as function of time. In addition, the americium sorption using radionuclide mixtures (e.g., Am-241 and U-232) and its desorption in the presence of EDTA was investigated by means of bath-type experiments. The experimental data (e.g., K_d values) revealed that the sorption efficiency depends on the MP type and the pH that governs the Am(III) speciation in the solution, and the PN6 surface charge determines the sorption efficiency. Moreover, the desorption of Am(III) from MPs is a time-dependent process, and the presence of complexing agents (e.g., EDTA) enhances the desorption efficiency. The K_d values in the studied water samples were relatively close to one another and similar to corresponding values in soil systems, indicating the significant role of microplastics on the behavior of americium in natural aquatic systems.

Keywords: americium; PE; PN6- microplastics; (de)sorption; K_d values; environmental waters



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1. Introduction

Microplastics are tiny plastic pieces (length < 5 mm) that are found in the environment as a result of the degradation of larger plastic pieces and/or commercial product development, and they are persistent pollutants, due to the slow degradation or their continuous release into the environment [1,2]. Furthermore, microplastics have relatively large specific surface area and can adsorb various pollutants, thus affecting their mobility and bioavailability and becoming a growing environmental issue on a global scale [1,3]. Environmental microplastics are usually detected and characterized with spectroscopic techniques, such as FTIR [4,5] and micro-FTIR [6]. The interaction of microplastics with metal ions is performed usually in batch-type experiments using ICP-MS [4,6], ICP-OES [5], and other atomic spectroscopy techniques for the metal/metalloid analysis, as well as radiometric methods (e.g., alpha spectroscopy and liquid scintillation counting) in the case of radionuclides [7–9]. Microplastics, which are omnipresent in the environment and have a remarkable dynamic behavior in atmospheric and aqueous environments, may act as contaminant vehicles transporting (radio) toxic substances along the food chain into larger organisms, including humans [10]. The contaminants can be organic and inorganic chemicals [11,12], pharmaceuticals [13], metal ions [14–17], and radionuclides. The latter is of particular interest because, even at very low concentrations, they can have deleterious effects, due to the emitted ionizing radiation. In this context, alpha-emitters are of particular importance and associated with their high ionization potential and biological damage [18]. Studies related to the (de)sorption of radionuclides by microplastics are necessary, in order to understand the migration of radionuclides in the geosphere and their accumulation in living organisms. However, there are only a few studies related to the interaction of radionuclides with microplastics and include the interaction of Cs-134, Sr-85 [19], I-129 [20], U-232 [7,9], Ra-226 [9], and Np-237 [8]. The sorption of the radionuclides by microplastics

is basically ascribed to weak (van der Waals) interactions [21,22], cation-dipole, electrostatic interactions [21,23,24], and complex formation between surface active moieties (e.g., carboxylate-, amide-groups, etc.) [7,8,21,22].

In this study, we have investigated the interaction of americium with microplastics. Americium is an artificial radioactive element; the longest-lived and most common isotopes of americium are ^{241}Am and ^{243}Am , and they have half-lives of 432.2 and 7370 years, respectively. Although not confirmed, trace amounts of americium may occur naturally in the uranium minerals produced by nuclear reactions. Nowadays, elevated americium can be detected in the areas used for nuclear weapons tests, as well as at the sites of nuclear incidents (e.g., Chernobyl). Generally, the average radioactivity concentration in surface soils is about 0.37 mBq/g [25]. In everyday life, americium (Am-241) is used in common type smoke detectors as a source of ionizing radiation. The amount of americium in a smoke detector is 37 kBq, and it is deposited in the form of americium dioxide. In addition, Am-241 has been used as ionizing source in lightning rods at radioactivity levels up to 212 MBq. Moreover, americium was used as portable gamma ray and Am-241, in combination with beryllium as a neutron source [26]. Am-241, with a half-life of 432.2 years, decays slowly into Np-237-emitting alpha particles and gamma radiation, which may be harmful for living organisms, including humans. The radioactive material from these devices can be released into the environment due to improper handling and disposal. The americium can be dissolved and enter the food chain; after ingestion, even in small amounts can cause cancer to human beings because of the increased radiotoxicity Am-241 and its accumulation in critical organs (e.g., kidneys, lungs, and bones). It is well-known that microplastics can interact with metal ions via different mechanisms, such as cation-dipole and electrostatic interactions [27], and affect their environmental behavior and bioavailability [28]. In this context, the interaction of Am(III) with microplastics is of particular interest, regarding their impact on the environmental behavior and bioavailability of americium.

The most stable oxidation state of americium in aqueous solutions is +3, and the Am(III) chemistry is similar to the chemistry of trivalent lanthanides, which are often used as non-radioactive analogues for Am(III) [29]. In aqueous solutions and under ambient conditions, Am(III)-hydroxo-carbonate ($\text{Am}(\text{CO}_3)(\text{OH})$) is the solubility-limiting species [30]. In acidic solutions ($\text{pH} < 7$), the Am^{3+} ion dominates the solution species. However, above pH and under ambient conditions, the carbonate complexes ($\text{Am}(\text{CO}_3)_n^{(2n-3)+}$) predominate in the solution, and the relative amount of the Am(III)-hydroxo complexes is generally below 10%. At pH 9, the $\text{Am}(\text{CO}_3)_3^{3-}$ species is the predominant species in the solution [31–33]. In the environment, americium is present in its trivalent oxidation state (Am(III)) and very unlikely to undergo redox reactions. The solubility limiting Am(III) species is the insoluble trivalent americium hydroxocarbonate, $\text{Am}(\text{OH})(\text{CO}_3)$, and hydrolysis and complexation with carbonate and dissolved organic matter (e.g., humic acids) determine its distribution and stability in aqueous solution. However, the interaction of the dissolved species with mineral and rock surfaces, as well as colloidal species, play a cardinal role, regarding the radionuclide's migration in the geosphere [34,35].

The present study deals with the interaction of americium with PN6 (polyamide nylon) [8,9] and PE (polyethylene) microplastics, which are produced in millions of tons annually and find application in several industrial sectors [36]. PN6 (polyamide nylon) is a polyamide polymer, with amide surface groups that may interact with polyvalent metal ions, including Am(III) [8,9]. On the other hand, PE (polyethylene) is a polymer with hydrophobic surface moieties, and the interaction between the PE surface and ionic species may occur through weak interactions [37]. Polyamide and polyethylene microplastics have been used in this study because the associated polymer materials are widely used in the industry and everyday life. Moreover, PN6 is a polar microplastic, and PE is a non-polar microplastic; thus, the comparison between these two microplastic materials will highlight the role of the microplastic composition, regarding the sorption affinity towards metal ions. The sorption affinity will be evaluated, in terms of the linear distribution coefficient (K_d), which is still a fundamental parameter, regarding the migration of radionuclides in the

geosphere and performance assessment studies. The effect of different physico-chemical parameters (e.g., pH, composition, and contact time) affecting the Am(III)-microplastic interaction, as well as the desorption of the radionuclide in de-ionized water and EDTA solutions, have been investigated by batch-type experiments. EDTA has been used in the present study, because this hexadentate iminodiacetic ligand is a strong complexing ligand and has been widely used in the nuclear industry and research labs for decontamination purposes [38].

2. Experimental

2.1. Materials and Analytical Methods

All experiments were performed under ambient conditions (23 ± 2 °C) in 20 mL polyethylene plastic containers. For the preparation of the reference and test solutions, standard tracer solutions of Am-241 (North America Scientific Inc., Los Angeles, CA, USA) and U-232 (National Physical Laboratory, Teddington, UK) were used, and their total activity was 12.05 and 4.923 Bq/g, respectively. The initial activity concentration of the sorption test and reference solutions was 25 Bq/L, which corresponds to molar concentrations of about 0.1 picomole and 1 picomole for the uranium and americium isotope, respectively. The two microplastics (MPs) under investigation were polyethylene (PE, 40–48 µm, Sigma-Aldrich, Darmstadt, Germany) in powder form and polyamide nylon granules (PN6, 5–50 µm, average 15–20 µm, GoodFellow, Hamburg, Germany). To study the sorption efficiency of the MPs for americium and uranium radionuclides, the experiments were carried out in artificial solutions using deionized water at different pH regions (pH 4, 7 and 9), simulated seawater at pH = 8.3, and in seawater collected from a coastal area in Cyprus. Desorption of Am-241 was performed in solutions with deionized water and 0.001 M EDTA aqueous solution. Analysis of Am-241 and U-232 was performed by using an alpha spectrometer (Canberra, Loches, France) after electrodeposition of the radionuclide's uranium onto stainless steel discs, as described elsewhere [39]. In addition, radiometric analysis was also performed using a liquid scintillation counter (Triathler, Hidex oy, Turku, Finland) after mixing 1 mL of the test solution with 19 mL of scintillator solution (Aqualight, Hidex, Turku, Finland) [40]. The experimental repeatability for all experiments/measurements was below the counting uncertainty, which was set to a maximum value of 10% for all measurements.

2.2. (De)Sorption Experiments

The sorption experiments of the Am-241 and Am-241/U-232 radionuclides was carried out as follows: 0.5 g of the MPs were added to 10 mL of the radionuclide solution in plastics 20 mL in screw cap containers. The activity for both radionuclides in the single radionuclide experiment and binary Am-241/U-232 system was 25 Bq/L, which corresponds to a molar concentration of about 0.1 picomole and 1 picomole, respectively. The solutions were shaken on a linear stirrer (SK-R1807, DLAB, Beijing, China) with a constant rate (45 min^{-1}) for 10 days contact time under ambient conditions. At certain time intervals, 50 µL of the solution were taken and the residual radionuclide concentration in the solution was determined by alpha spectrometry and liquid scintillator counting (LSC). The alpha spectrometer was calibrated using a standard reference solution (1.02 Bq/mL, U-232 standard reference solution) and calibration mixed source (U-238/234, Pu-239, and Am-241), with a total activity of 6.6 Bq (Eckert & Ziegler), while the LSC calibration was performed using standard reference solutions. The distribution coefficient, K_d was determined as reported elsewhere [9]

$$K_d = C_{ads}/C_{aq} \text{ (L/kg)} \quad (1)$$

where C_{ads} (Bq/g) is the Am-241 sorbed by the MP, C_{aq} (Bq/L) is the concentration of Am-241 in solution. The amount of americium sorbed by the MPs is calculated by subtracting the amount of americium sorbed on the walls of the PE bottle from the total amount of americium adsorbed.

Am(III) sorption experiments under similar conditions have been also performed in simulated and natural seawater samples. The simulated seawater has been prepared by dissolving 23.926 g/dm³ NaCl, 1.519 g/dm³ CaCl₂·2H₂O in 1 L de-ionized water, and the composition of the natural seawater is described elsewhere [41].

Desorption experiments were performed by contacting americium-loaded MPs, which were obtained from the sorption experiments, with 10 mL of deionized water (pH 6.6) or 0.001 M EDTA aqueous solution (pH 4.5). The contact time of the microplastics with the solution was 10 days and concentration of Am-241 activity in the solution was determined by alpha spectroscopy and liquid scintillation counting, in order to evaluate the amount of Am-241 desorbed. In addition, to account for the interaction of the radionuclides with the container walls, similar (de)sorption experiments (without MPs) were performed.

3. Results and Discussion

3.1. pH and Contact Time Effect on the Am-241 Sorption by PN6 and PE

The sorption of trivalent americium (Am(III)) on the microplastic surfaces depends on the metal ion speciation, as well as their surface active sites and charge. Am(III) is the only thermodynamically stable oxidation state of americium in aqueous solutions under ambient conditions. The Am(III) species that govern the americium chemistry in aqueous solutions under ambient conditions are Am³⁺ in the acidic pH region (pH < 7), Am³⁺, Am(OH)₂⁺ and Am(CO₃)⁺ in the near neutral pH region (7 < pH < 8), and Am(CO₃)₂⁻ and Am(CO₃)₃³⁻ in the alkaline pH region (pH > 8) pH 9 [31–33]. On the other hand, the surface of the PE microplastics is expected to be negatively charged for pH > 2.5, due to the dissociation of carboxyl groups present on the PE surface, as a result of its partial oxidation during polymerization and further processing [42]. The presence of carboxylic moieties on the PE surface is expected to attract and bind cationic species through the formation of inner sphere complexes between the cationic metal species and carboxylic surface group. In addition, PE can bind cations through weak cation-dipole interactions between the cations and instantaneous C-H dipoles on the polymer surface. Regarding PN6, the overall surface charge of this microplastic is positive up to pH 4 and becomes negative for pH > 4 [43]. This is attributed to the presence of the amide and amino groups, which are protonated under acidic conditions. In addition, carboxylic moieties, which are present due to partial degradation and oxidation of the polymeric material, start dissociating for pH > 2.5. Moreover, the cations can interact via cation-dipole interaction with the carbonyl moieties of the amide group and amino groups present on the PN6 surface.

The sorption efficiency of Am-237 by PN6 and PE microplastics in the acid, neutral and alkaline pH region has been investigated as a function of time, and the corresponding data are graphically presented in Figure 1. From the data, it is obvious that the sorption of Am(III) differs significantly from one microplastic to another, and this can be ascribed to the amide/amino moieties, which are present on PN6, depending on the solution pH determine the surface charge. Regarding PE, the pH does not significantly affect both, i.e., the sorption efficiency (K_d values) and sorption kinetics. This is because, within the investigated pH range (4 ≤ pH ≤ 9), the sorption is mainly based on the interaction of the partially oxidized surface (carboxylic moieties) and induced dipole interactions between the PE and Am(III) species, and the carboxylic moieties are almost quantitatively dissociated for pH ≥ 4. On the other hand, the sorption kinetic is basically determined by the diffusion of the Am(III) species towards the microplastic surface because of the very low americium concentration [9].

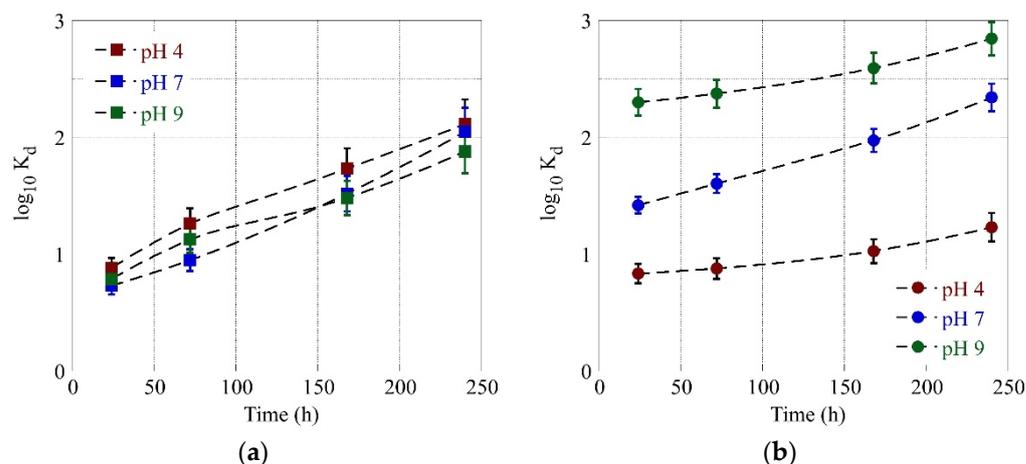


Figure 1. K_d values for the Am(III) sorption by (a) PE and (b) PN6 microplastics, as a function of pH and contact time.

On the contrary, the Am(III) sorption by PN6 is strongly affected by the pH, and the highest sorption efficiency (K_d value) was observed for pH 9, followed by pH 7 and pH 4. This is mainly attributed to the presence of deprotonated amino and carboxylic present on the PN6 surface, which compete Am(III) carbonate complexation and form possibly ternary Am(III) surface complexes. At pH 7, the sorption efficiency declines, due to the protonation of amino groups, which repel the positive Am(III) species (Am^{3+} , $\text{Am}(\text{OH})_2^+$ and $\text{Am}(\text{CO}_3)^+$) and destabilize Am(III) surface binding. Similarly, at pH 4, a further decline of the K_d values is observed because Am^{3+} is the predominant species in solution and the positive surface charge increases further. Regarding the sorption kinetics, there is almost no effect because the sorption kinetics are mainly limited by the Am(III) species diffusion towards the PN6 surface because of the extremely low concentration (picomole range) of Am-241 [9].

A comparison of the K_d values obtained for the two different microplastic types reveals that, at pH 9, PN6 possesses the highest sorption affinity for Am(III), with the maximum $\log K_d$ (L/kg) value determined to be 2.8. Additionally, the $\log K_d$ value associated with PN6 at pH 7 (after 250 h) was significantly higher than the $\log K_d$ (L/kg) values obtained for PE (pH 7), equal to 2.3. On the other hand, the $\log K_d$ values determined for PN6 at pH 4 were lower than the corresponding values for PE, and the maximum value (after 250 h) was equal to 1.2 L/kg. Regarding the Am(III) sorption by PE, the highest $\log K_d$ (L/kg) values obtained were close to 2.0 ± 0.1 . Considering the $\log K_d$ determined for the U(VI) sorption by PN6 ($\log K_d$ (L/kg) = 3.4) and PE ($\log K_d$ (L/kg) = 2.5 ± 0.1) [9], it is obvious that the sorption affinity of the two microplastics was significantly higher for U(VI) than for Am(III) in the near neutral and alkaline pH range.

3.2. Simultaneous Am-241 and U-232 Sorption by PN6 and PE as Function of Time

In order to have a direct comparison of the affinity of PN6 and PE for Am(III) and U(VI), the sorption experiments were carried out with a U-232 and Am-241 mixture solution, and the corresponding results are graphically presented in Figure 2. According to the data shown in Figure 2, the highest sorption capacity of PN6 was observed for U(VI), with the lowest for Am(III). This is in agreement with sorption affinities (K_d values) values observed for the single radionuclide systems [9]. On the other hand, the significantly different behavior between the two actinide cations (Am^{3+} and UO_2^{2+}) can be attributed to additional interactions, which may occur between the uranyl cation (UO_2^{2+}) and protonated PN6 surface amide or amino moieties. These interactions probably include hydrogen bonding (e.g., $(>\text{N}-\text{H}^+ \cdots \delta^-\text{O}=\text{U}=\text{O})$) and have also been observed in other, similar systems [39]; they result in increased stabilization of the formed U(VI) species. This host-guest type hydrogen bonding is possible only for actinyl cations that enhance their chemical affinity

towards amide or iminoacetic compounds [9,39]. A schematic illustration of the respective interactions is provided in Figure 3. On the other hand, the sorption efficiency of both actinide cations by PE is almost similar, indicating similar interactions between the actinide cations and PE surface.

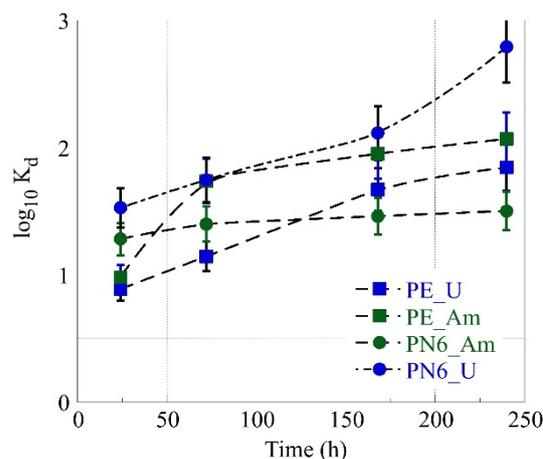


Figure 2. K_d values for the Am(III) and U(VI) sorption by PE and PN6 microplastics from a Am-241 and U-232 mixture solution, as a function of contact time at pH 4.

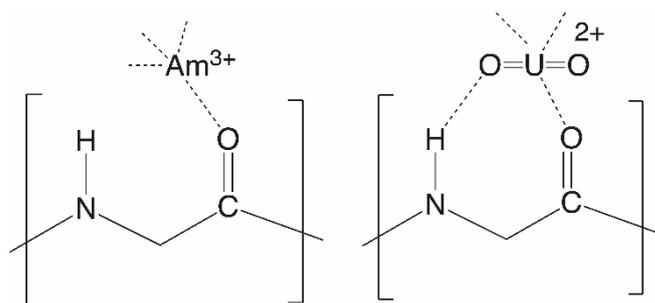


Figure 3. Schematic illustration of the interactions between Am(III) or U(VI) cations and the PN6 amide moiety.

3.3. Am-241 Sorption by MP's in Environmental Water Solutions

In order to study the chemical affinity between the two microplastics (PN6 and PE) and Am(III), under environmentally relevant conditions, sorption experiments were carried out in simulated and natural seawater samples. The results obtained are graphically summarized in Figure 4 and indicate that the K_d values determined in the seawater samples were close to the corresponding values obtained under weak alkaline pH conditions ($7 < \text{pH} < 9$). In addition, the sorption kinetics in natural seawater seems to be slower, whereas the sorption efficiency of PN6 was similar in simulated and natural seawaters. Moreover, the sorption efficiency of PN6 for Am(III) was higher, compared to PE. Specifically, the K_d values for Am(III) by PN6 and PE for simulated and natural seawater samples after 250 h of contact time were: $\log K_d$ (sim SW, L/kg) = 2.5 ± 0.1 , and $\log K_d$ (sim SW, L/kg) = 2.1 ± 0.1 , and $\log K_d$ (SW, L/kg) = 1.9 ± 0.1 , respectively. These values were close to the corresponding values determined for the U(VI) sorption by the two microplastic types in natural seawaters [9]. In addition, the K_d values determined for the sorption of Am(III) by the PN6 and PE microplastics were close to the best estimate of the linear distribution coefficient ($\log K_d$ (L/kg) = 3.7) evaluated and within the range of K_d reported for Am(III) in soils [44], indicating the possible impact of microplastics on the geochemical behavior and bioavailability of Am(III).

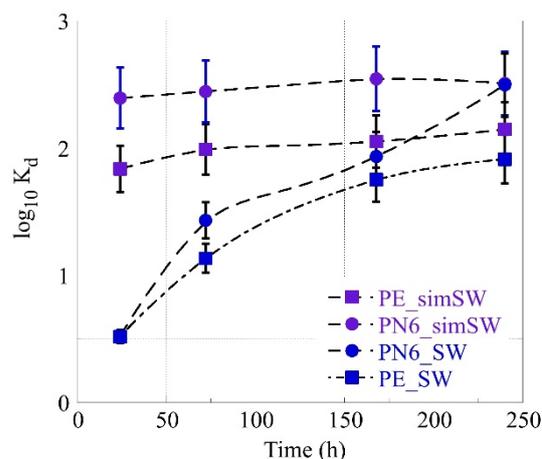


Figure 4. K_d values corresponding to the Am(III) sorption by the studied MPs in seawater and simulated seawater.

3.4. Desorption of Am-241 from MP's in Deionized and EDTA Water Solutions

The relative desorption of Am(III) from the PN6 and PE microplastics were investigated in de-ionized water (pH 4) and 0.001 M EDTA aqueous solution (pH 4.5). Figure 5 shows the %-relative desorption of Am(III) from the studied micro-plastics and in the different media, as a function of time. According to the data shown in Figure 5, it is obvious that the desorption is a time-dependent sorption, and the Am(III) desorption is more effective in the presence of EDTA, due to the stabilization of Am(III) in solution in the form of Am-EDTA complexes [45]. The Am-EDTA complexes are very stable ($\log K_f \sim 16$) [38], increase Am(III) concentration in solution, and restrict its sorption on the microplastic surface.

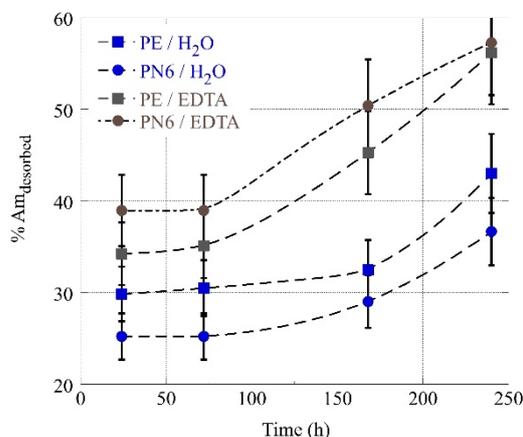


Figure 5. Relative amount of Am(III) desorbed from PE and PN6 microplastics in laboratory deionized and 0.001 M EDTA solutions, as function of time.

4. Conclusions and Future Work

Microplastics appear to retain Am-241, even in the picomolar concentration range. The degree of adsorption depends on the solution pH and Am(III) speciation. According to the K_d values obtained, maximum adsorption capacity occurs at pH 9, followed by pH 7 and pH 4. The K_d values associated with the Am(III) sorption by PN6 were higher than the K_d values corresponding to PE, due to the amide/amino moieties, which most probably favor the formation of ternary Am(III) surface complexes, particularly in the alkaline pH range (pH 9). The difference in the K_d values of the Am^{3+} and UO_2^{2+} cations is attributed to additional interactions that the uranyl oxygens can develop with PN6, while, for PE, there was no significant difference, regarding the K_d values for both actinides. In complex systems (e.g., in natural seawater), the adsorption kinetics are slower than in de-ionized

water and simulated seawater. Nevertheless, the K_d values corresponding to PN6 were similar in simulated and natural seawater samples. Regarding the desorption of americium from the studied microplastics, the desorption is a time-dependent process, and it is favored in the presence of EDTA.

Future studies could focus on the sorption of other environmentally relevant radionuclides, experiments with altered microplastic samples (e.g., weathered and biofilm coated microplastics), and sorption studies from multi-radionuclide solutions and soil/sediment-microplastic mixtures.

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