Metallurgical and Materials Data 2, no. 3 (2024): 103-107

Publisher: Association of Metallurgical Engineers of Serbia

Metallurgical and Materials Data

www.metall-mater-data.com

Electrospun MOF-loaded polyamide membranes for Y^{3+} radioisotopes removal

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ARTICLE INFORMATION:

https://doi.org/10.56801/MMD39

Received: 28 October 2024
Accepted: 20 November 2024

Type of paper: Research paper

ABSTRACT

The environmental impact of radionuclides, found in nuclear waste, nuclear power plants, agriculture, industrial effluents, research centers, and nuclear medicine facilities, is highly important, especially in the emerging nuclear era. Prior to its stabilization and permanent disposal, the development of appropriate techniques for radionuclide removal from the environment is a critical topic. Particular attention is given to materials that are easy to synthesize, manipulate, and demonstrate high pollutant removal efficiency. Among different radionuclides, yttrium isotopes are one the most common products associated with nuclear power activities. Polyamide (PA) and PA-MOF (MOF - metal-organic framework) nanofibrous composite membranes (containing 1% and 10% MOF), obtained via electrospinning, were investigated as sorption materials for yttrium ions. The highest removal efficiency of 76% was achieved using PA from simulated seawater samples at pH 5.7. Microstructural and morphological characterization of the prepared membrane samples confirmed the existence of both crystalline and amorphous phases of polyamide and wrinkled fiber arrangement with a diameter of less than 0.5 μm. Agglomerates of MOF particles, ranging in size from 2 to 8 μm, are embedded between the PA fibers. Changes in the lattice vibrations corresponding to the CH^{2-} groups, observed in the range 1420-1475 cm^{-1}, indicated that hydrogen bonding interactions were favorable for the sorption process of yttrium ions on the prepared materials.

Keywords: polyamide; metal organic framework; nanofibrous membranes; radionuclide removal; yttrium

1. Introduction

Revival of nuclear energy as a clean energy source and wide application of nuclear resources in different fields may, besides the benefits, also represent a threat to the environment. The environmental impact of radionuclides, which can be found in nuclear waste, nuclear power plants, agriculture, industrial effluents, research centers, and nuclear medicine facilities is significant and becoming more important in the new nuclear era (Gendy et al. 2021). Developing appropriate techniques for radionuclide removal from the environment is a very important topic (prior to its stabilization and disposal), and attention is paid to the materials that are easy to synthesize and with high pollutant removal efficiency. Nuclear facilities are usually located near the seashore, so in case of any leaking or nuclear accident, such as the one in the Fukushima plant, a lot of radioisotopes may be dispersed in the surrounding sea area (IAEA 2024).

Among different radionuclides, the most common products related to nuclear power activities are yttrium isotopes. Yttrium has one non-radioactive isotope, ^{89}Y, and its toxicity varies depending on the compound. The most important isotopes of yttrium are ^{91}Y and ^{90}Y, with half-lives of 58.51 days and 64 hours, respectively. Yttrium-90 (^{90}Y) is a radioactive isotope, a beta emitter, widely used in nuclear medicine, particularly for targeted cancer therapies such as radioembolization and radioimmunotherapy (Hosono et al. 2019; Tong et al. 2016; Puvvada et al. 2018). Although ^{90}Y disposal is regulated, and its short half-life of 2.67 days generally minimizes environmental risk, improper disposal or nuclear accidents can still result in localized environmental contamination (Hosono et al. 2019). Yttrium-91 (^{91}Y), beta and gamma emitter is a byproduct of nuclear fission and can be found in nuclear waste, and used for trace analysis (Gilligan et al. 2024; Pierson et al. 2022). Due to its relatively long half-life of 58.51 days, this isotope represents a greater environmental risk than ^{90}Y (Gilligan et al. 2024). The removal of both isotopes from contaminated areas is typically performed by sorption, ion exchange, and other radionuclide removal techniques.

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Numerous materials have been investigated for the sorption of radionuclides from (simulated) waste solutions under different experimental conditions. Removal of yttrium ions by sorption usually includes recovery from wastewater including other rare earth elements (scandium and 15 lanthanides) (Z. Li, Zhu, and Yao 2024), but recent research has focused on yttrium as a primary target for removal. To date, a limited number of studies have investigated the removal of yttrium ions by sorption, and materials used are diverse, including calcium and sodium alginates (Khotimchenko et al. 2015), Purolite C100Na Resin (Hamid and Nasab 2021), titanium dioxide (Vasylyeva et al. 2021; Mironyuk et al. 2023), among other materials (Mahmoud and Mohamed 2019). In 2023, Mironyuk et al. investigated sorption of yttrium by the sodium-modified titanium dioxide (Na-TiO_2). The sorption capacity was high - about 259 mg/g, (Mironyuk et al. 2023), higher than unmodified TiO_2 (7 mg/g), or other modifications - 4As–TiO_2 (127 mg/g) and Nd/4As–TiO_2 (24.8 mg/g) (Vasylyeva et al. 2021). Electrospinning emerged as a polymer processing technique which enables the production of various composite materials with improved properties and wider application. The electrospinning is a nanofiber-forming technology in which continuous filament of a polymer liquid is drawn by a high electrostatic force through a spinneret and then deposited randomly on a grounded collector (Nirmala et al. 2010). Due to their specific properties, such as good thermal and mechanical stability, high surface area, sorption capacity, etc., electrospun polymer nanofibers, particularly polyamide and their composites are used in different areas such as wastewater treatment, biomedical applications, energy storage (Sobolčiak et al. 2017; Matulevicius et al. 2014; Rist and Greiner 2024; Zhu et al. 2024; Zhang et al. 2022; Laudenslager, Scheffler, and Sigmund 2010; Jelmy et al. 2024; Lu, Murray, and Zhu 2019; Luraghi, Peri, and Moroni 2021; Selvakumar et al. 2012; Cui et al. 2020).

Electrospun polymer nanofiber membranes in general are used for removal (mostly by filtration and sorption) of various pollutants, such as oils, heavy metals, or dyes from the wastewater (Cui et al. 2020; Radoor et al. 2024; Mowafi and Tohamy 2024). The advantage of these materials is that they are easier to manipulate and remove from the medium than powdered materials, while the great surface and porosity enable high efficiency. Active materials-loaded polymer nanofibers are achieving better performance, and their activity can be tailored according to the end-user demand. Active agents that are used are very diverse, such as basalt (El-Sheikh et al. 2023), nanoclay and TiO_2 (Aydin-Aytekin et al. 2022), cellulose (Sobolčiak et al. 2017), zeolite (Hosseini et al. 2024), chitosan (Nirmala et al. 2011), etc. depending on the intended application. Those fibers were used for the removal or extraction of various radionuclides, such as the uranium extraction from seawater (Jiang et al. 2022) or the removal of Cr^{3+}, Co^{2+}, and Cs^{+} from aqueous solutions (Essalhi et al. 2024). Similar to electrospun polymers and polyamides, MOFs (such as UiO-66) are also applied in the sorption and removal of different radionuclides, such as UO_2^{2+}, Th^{4+}, TcO_4^{-}, ReO_4^{-}, Sr^{2+}, Eu^{3+}, Cs^{+}, Co^{+} Se^{4+}, Se^{6+}, etc. (Gendy et al. 2021; Wang and Xu 2023)

To our knowledge, no research has been published on the removal of yttrium ions from aqueous solution by using electrospun polyamide, UiO-66 or their composites. Removal of radioisotopes, such as yttrium, using powdered materials may be difficult, so one of the ways of removal may be by using highly porous nanofibrous membranes which can be easily manipulated, e.g. put and removed from the seawater. Due to its short half-life, $^{90}Y^{3+}$ was used for the investigation of sorption properties from synthetic seawater using synthesized polyamide-based membranes.

2. Methods

Polyamide (PA) nanofibers and PA nanofiber composites with UiO-66 in different weight ratios were prepared by electrospinning method. For the production of PA nanofibers, PA-6 Akulon®, obtained by local industrial partner, was used.

UiO-66 xerogel was synthesized at room temperature by mixing terephthalic acid, zirconium oxychloride octahydrate and methanol, centrifugation of obtained white suspension, washing with dimethylformamide and methanol, and then dried at room temperature overnight (F. Li and Wu 2021).

Preparation of solutions for electrospinning

In the first step, neat PA-based solutions containing 20% of PA were prepared by mixing in formic acid for 24 hours at room temperature. Prior to electrospinning, MOF was added to the solutions in amount of 1 and 10%, and mixed for 1 h. Three solutions were prepared in total, one neat PA solution as control and two composite solutions.

Electrospinning process

For the preparation of nanofibrous membranes, Fluidnatek LE-10 electrospinning instrument (Bioinicia, Spain) with two syringe pumps (with 10,000 μLh^{-1} maximum feed rate) and high voltage supply (with a maximum voltage of 30 kV) was used. The feed rate was 500 μLh^{-1}, with voltage of 20-22 kV and distance of 10 cm.

Before the removal of radionuclides, membranes were cut into 1x2 cm rectangular shaped samples, in order to be easily applied and removed from the solution. Depending on the wt.% of MOF in material, nanofibers are denoted as PA (0 wt.% of MOF), PA-MOF1% (1 wt.% of MOF), and PA-MOF10% (10 wt.% of MOF).

Sorption experiments

Batch experiments were conducted to evaluate the efficiency of electrospun PA and PA/UiO-66 nanofibers in removing $^{90}Y^{3+}$ radionuclides from water solutions.

The efficiency of ^{90}Y removal was determined by inserting nanofibers samples in 2 mL of sea water (35 ppm or 3.5% NaCl) pH 5.7 and 8.1 with addition of 0.01 mL solution of ^{90}Y (activity 0.74MBq). After some period (1 hour to 24 hours) nanofibers samples were removed, and radioactivity was measured in the remaining solution and nanofiber samples on the gamma counter Perkin Elmer Wizard 1480 for 20 sec.

Microstructural and morphological characteristics

The microstructure of the electrospun membranes was examined by X-ray diffraction (XRD) technique using a Rigaku Ultima IV diffractometer, with Cu Kα radiation (λ = 1, 4178 Å). The diffractograms were collected in the 2θ range from 10 to 50° with the scan rate of 5 °/min.

The morphology of the samples was analyzed using scanning electron microscopy (SEM) by JEOL JSM 6460 LV Oxford INC (Tokyo, Japan).

Infrared spectra were obtained using a Fourier transform infrared spectrophotometer (FTIR) the Spectrum Two Spectrometer (PerkinElmer Inc., USA) in the range 4000–450 cm^{-1}, with a resolution of 4 cm^{-1}.

3. Results and discussion

3.1. Sorption performance of $^{90}Y^{3+}$ on PA and composites PA-MOF1% and PA-MOF10%

In order to determine the possibility of finding an effective and easy way to remove radioactive $^{90}Y^{+3}$ spilt and dispersed in the seawater after the potential incident, sorption performances of electrospun membranes made of polyamide and its PA-MOF composites were investigated. Electrospun membranes may represent a scaled-up way for easy removal of radioactive isotopes from the sea. Removal efficiencies of $^{90}Y^{3+}$ using PA, PA-MOF1% and PA-MOF10% composites after 1h, 3h and 24h of contact time on pH 5.7 and 8.1 are presented in Table 1. Sorption was performed in saline solution due to many nuclear reactor plants being built on the seacoasts. As can be seen from Table

1, pure PA showed the highest sorption efficiencies compared to both PA-MOF composites and slightly higher values in acidic solution, on pH 5.7. This indicates that non-woven electrospun PA membranes can be an excellent candidate for the removal of radioisotopes of Y^{3+} from seawater. Due to high ability of agglomeration of MOF, the number of active places along the fibers was decreased, and that might be the reason of lower activity of composite membranes.

Table 1. Removal efficiencies of $^{90}Y^{+3}$ sorption on PA and composites PA-MOF1% and PA-MOF10% after 1h, 3h and 24h on pH 5.7 and pH 8.1

Sample	Removal efficiency (%)					
	pH = 5.7			pH = 8.1		
	1 h	3 h	24 h	1 h	3 h	24 h
PA	49.63	62.84	76.58	59.66	64.25	70.53
PA-MOF1%	52.47	58.55	74.23	46.39	49.01	53.98
PA-MOF10%	43.86	61.67	72.91	46.61	47.24	51.55

3.2. *Microstructural and morphological characteristics of the PA and composites PA-MOF1% and PA-MOF10% electrospun membranes*

Morphology of the pure PA and both PA-MOF1% and PA-MOF10% composites are shown in Figure 1. PA exhibits a wrinkled fiber structure with a diameter of less than 0.5 μm. As described in the literature (El-Sheikh et al. 2023; Topuz et al. 2021) densely packed electrospun fiber structure provides high surface area and porosity, which further enable easy penetration of water molecules. As presented in Fig.1, both PA-MOF composites exhibit the same porous structure as pure PA, the same diameter and wrinkled fibers arrangement characteristic for electrospun membranes (Xue et al. 2019; El-Sheikh et al. 2023), with the agglomerates of MOF particles embedded within the fibers. UiO-66 particles have an irregular shape, in the range of sizes from 2 to 8 μm, and are not well dispersed within the fiber structure. Comparing the morphology of both composites, 10% of MOF compared to the sample with 1%, there was no change in the size, distribution or shape of MOF particles, nor the change in nanofiber morphology.

Diffractograms of the pure PA, PA-MOF1% and PA-MOF10% are presented in Figure 2. The broad peak at the diffractogram of the electrospun PA fibers may be the result of several factors: a) peaks overlapping in polymers is very common, b) PA has two crystalline phases as well as amorphous phase, all positioned very close (described in more detail below), and c) rapid solidification that occurs in electrospinning process can limit the development of crystallinity due to the lack of time to macromolecular chains to form crystalline forms (Razavizadeh and Niazmand 2020).

Namely, Shan et al. showed that PA can crystallize into two polymorphic forms, monoclinic α and pseudo-hexagonal γ forms, with the α form as a predominant form (Shan et al. 2005). The molecular chains of PA molecules are organized in parallel laminas with hydrogen bridges arranged in parallel chain arrangement making the γ phase of PA, or hydrogen bridges organized in antiparallel chain arrangement creating the α-crystalline phase of PA (Shan et al. 2005). The diffractogram of the PA shows a semi-crystalline structure, Fig. 2 a. Diffraction peaks that appear at 2θ = 20.4° and 23.78° are two distinctive features of the α-crystal form of PA, originating from the reflections of the (200) and (202) crystal planes, respectively (Shan et al. 2005). A peak at 2θ = 21.88° originates from the γ-crystalline form of PA that correspond to the reflection of the (001) crystal plane.

Reduction of the intensities of both crystal phases of PA can be noticed in the samples of the composites compared to neat PA nanofibers. So, even both, α and γ crystal phases of PA, peaks of the PA still exist around 2θ = 21.88°, a significantly pronounced amorphous structure characterizes the PA and PA-MOF composites.

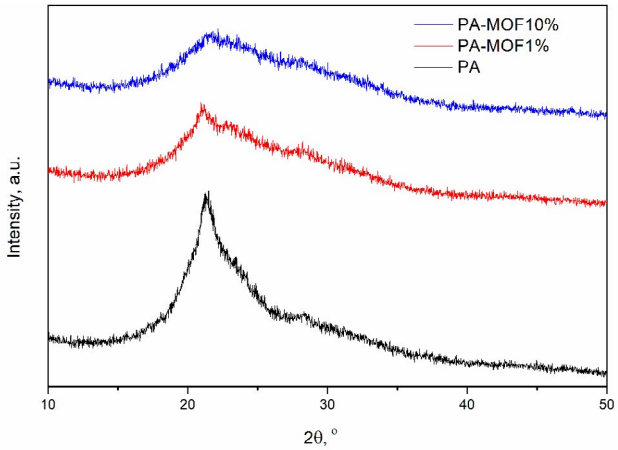

Fig. 2. Diffractograms of electrospun nanofibers PA, PA-MOF1%, and PA-MOF10%.

FTIR spectra of PA, PA-MOF1% as well as PA and PA-MOF1% after sorption of $^{90}Y^{3+}$ ions are presented in Figure 3. The wide band at 3400 cm^{-1}, originate from –OH stretching vibration of the absorbed water molecules. Bands located at 3304 and 1535 cm^{-1} can be assigned to N-H stretching and bending vibration of the hydrogen bonds in the amide II, respectively (Farias-Aguilar et al. 2014). C-H stretching vibrations in $-CH_2$ and $-CH_3$ groups were observed with a maxima at 2934 cm^{-1} and 2863 cm^{-1}, respectively (Farias-Aguilar et al. 2014). Further, N-H bond vibrations of amide I and II modes are responsible for the appearance of strong bands in the range of 1500 to 1700 cm^{-1} (Khori et al. 2020). Bands that appear in the region from 1420 to 1470 cm^{-1} belong to CH_2 bend vibrations from both crystalline and amorphous phases, while bands that appear at 1205 cm^{-1} originate from wagging CH_2 vibrations in the crystalline α phase (Porubská et al. 2012). Vibration in the $-CH_3$ group appears at 1372 cm^{-1} (Farias-Aguilar et al. 2014). Bands at 1124 cm^{-1} and 1205 cm^{-1} correspond to C-CO stretching in the amorphous phase, while peaks at 930 cm^{-1} correspond to the C-Co stretching in α and β crystalline phases. Characteristic bands of the α-phase of PA appear at 964, 959 and 928 cm^{-1}, while $-(CH_2)_n-$ chains are responsible for the band appearing at 730 cm^{-1} (Farias-Aguilar et al. 2014).

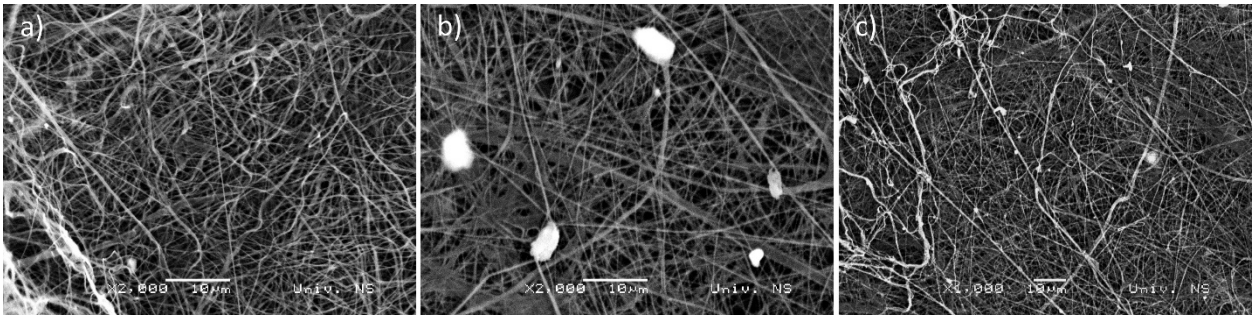

Fig. 1. SEM images of electrospun nanofibers: a) PA, b) PA-MOF1%, c) PA-MOF10%.

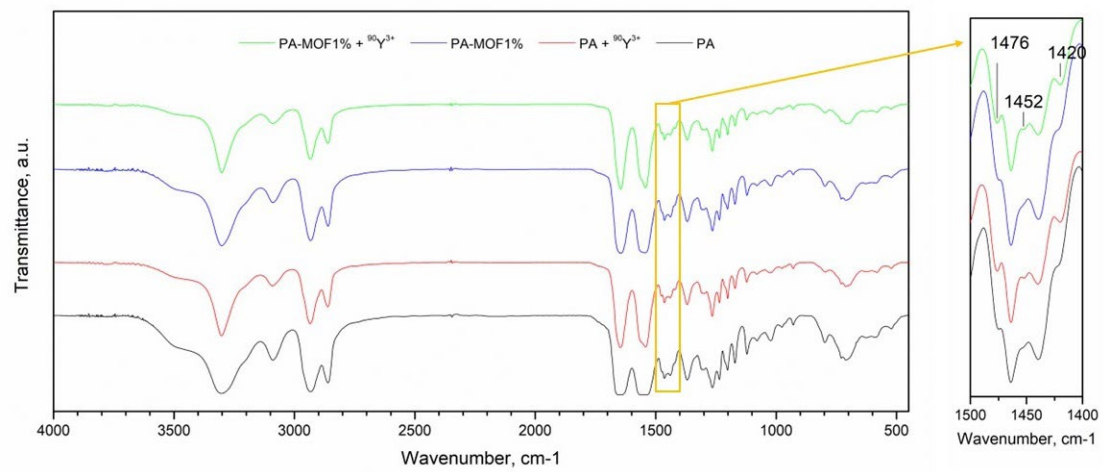

Fig. 3. FTIR spectra of PA, PA-MOF1% as well as PA and PA-MOF1% after sorption of $^{90}Y^{3+}$.

A variety of functional groups present at the surface of the pure PA as well as the PA-MOF composites indicate their potential to be used as sorption medium for yttrium ions. Khori et al. (Khori et al. 2020) proved the previously stated assertion of Xu et al. (Xu et al. 2015) that hydrogen bonding interactions may be favorable for the sorption process. Further, Porubská et al. (Porubská et al. 2012) showed that changes in the region from 1420 to 1440 cm^{-1}, where both crystalline and amorphous parts of the polymer coexist, can interact with protons. Changes in the FTIR spectra of PA and PA-MOF1% composite upon sorption of yttrium ions were observed in the region from 1420 to 1476 cm^{-1} indicating the place of its bonding.

4. Conclusion

PA and PA-MOF nanofiber composites (with 1% and 10% of UiO-66) were obtained by electrospinning. The results showed that non-woven electrospun PA and its MOF composites membranes may represent excellent candidates for the removal of radioisotopes of Y^{+3} from simulated seawater. The highest removal efficiency of 76% was obtained for PA at pH 5.7.

Microstructural and morphological characterization of the nanofibrous membranes confirmed the existence of both crystalline and amorphous phases of polyamide and wrinkled porous fiber structure with a diameter less than 0.5 μm in all samples. Agglomerates of MOF particles, sizes ranging from 2 to 8 μm, are embedded between the PA fibers. Changes in the lattice vibrations corresponding to vibrations of the CH_2- groups, which appeared in the range 1420-1475 cm^{-1}, indicated possible twisting and rotational deviation of hydrogen bridges in PA and PA-MOF composites by sorption of yttrium ions, indicating the place of yttrium bonding to the nanofibers. The further development of MOF dispersion within PA might result in higher efficiency of composite nanofibrous membranes.

Acknowledgments:

The research was funded by The Ministry of Science, Technological Development and Innovation of the Republic of Serbia under registration number: 451-03-66/2024-03/200017.

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