

DEGRADATION OF RESINS IN EPICOR-II PREFILTERS FROM THREE MILE ISLAND^a

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ABSTRACT

The Low-Level Waste Data Base Development--EPICOR-II Resin/Liner Investigation Program funded by the U.S. Nuclear Regulatory Commission is investigating the chemical and physical conditions of the synthetic ion exchange resins contained in several EPICOR-II prefilterers. Those prefilterers were used during cleanup of contaminated water from the Three Mile Island Nuclear Power Station after the March 1979 accident. This paper summarizes results and analyses of the second sampling of resins from prefilterers PF-8 and -20. Results are compared with baseline data from tests performed on unirradiated resins supplied by Epicor, Inc. to determine if degradation has occurred due to the high internal radiation dose. Results also are compared with results from tests performed on resins obtained from the first sampling of those two prefilterers.

INTRODUCTION

This paper discusses the resin degradation study conducted on organic ion exchange resins removed from two EPICOR-II prefilterers used during cleanup of contaminated water from Unit 2 of the Three Mile Island Nuclear Power Station. PF-8 (containing organic resins) and PF-20 (containing organic resins and zeolite) were selected for the resin degradation studies because they are highly loaded representatives (1400 and 2000 Ci, respectively) of the two types of EPICOR-II prefilterers. Descriptions of the prefilterers and the research being conducted can be found in Refs. 1, 2, and 3.

As part of the EPICOR and Waste Research and Disposition Program sponsored by the U.S. Department of Energy at the Idaho National Engineering Laboratory (INEL), 50 prefilterers were transported to INEL for temporary storage and preparation for disposal.^{4,5} Four prefilterers are being retained at INEL for studies funded by the U.S. Nuclear Regulatory Commission under the Low-Level Waste Data Base Development--EPICOR-II Resin/Liner Investigation Program. Those prefilterers are kept in temporary storage casks outside the Hot Shop of Test Area North Building 607 at INEL.

Resin degradation studies traditionally have been conducted using resins irradiated by an external source, such as a reactor core or Co-60 source. The gamma dose provided by an external source simulates the one received from radionuclides retained on the resin matrix by ion exchange. Modes of degradation do not differ between external and internal radiation; however, the literature notes that internal radiation causes more extensive damage than external radiation, presumably from short-range, high-energy beta radiation. The EPICOR-II resins had been contained in the prefilterers for approximately six years and experienced internal radiation doses of over 10^7 rad.

Throughout this paper the following nomenclature applies to various sizes and configurations of materials removed from the EPICOR-II prefilterers for examination:

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- Resin Core--One core was removed from each prefilter (PF-8 and -20) using coring tools.
- Resin Samples--100-mL volumes removed from the resin cores. Three samples (PF-8#1, PF-8#2, and PF-8#3) were removed from the PF-8 core. One sample (PF-20) was removed from the PF-20 core.

MATERIALS AND METHODS

To develop baseline data for the resin degradation studies, unirradiated ion exchange resins representative of those in the EPICOR-II prefilterers were provided by Epicor, Inc. The unirradiated resins were identified by functional group, exchangeable species, and matrix (e.g., sulfonic acid, strong acid cation, and styrene). They were characterized for comparison with the irradiated resins obtained from prefilterers PF-8 and -20. Both the unirradiated and irradiated resins were examined, using similar techniques. ASTM tests were used to determine moisture content, density, salt-splitting capacity, and exchange capacity.⁶ Infrared spectroscopy was used to identify functional groups (e.g., sulfonic acid, phenolic, and quaternary ammonium) and matrix material (styrene and phenol). Vapor phase chromatography was used to analyze the rinse and soak solutions from the ASTM tests quantitatively for styrene and divinylbenzene. Barium chloride precipitation was used for determining sulfonic acid groups, and scanning electron microscopy for determining the physical condition of the resins.

Coring

Resin cores were removed remotely from EPICOR-II prefilterers PF-8 and -20 in 1983 and 1985, using coring equipment based on a design developed at Battelle Columbus Laboratories and modified for use at INEL.⁷ The coring equipment consists of the following: (a) coring tool guide, which locates the coring tool over the prefilter resin bed; (b) spacer, which aligns and guides the coring tool into the bed; (c) coring tool and shutter, used for collecting, transporting, and storing the resin core; (d) casing and shutter, used for retaining the void space in the bed and preventing collapse of the resin bed after removal of the coring tool; and (e) vibrator tool, which drives the coring tool, casing, and shutters into the resin bed. Reference 2 further describes the coring equipment.

Gamma Scanning

Full-length isotopic gamma scans were made of each resin core to produce an axial profile of radionuclide distribution. Those profiles were used to determine the regions of highest radionuclide loading in the cores. Then, isotopic spectral gamma measurements were obtained at locations of highest radionuclide concentration.⁸ The two radionuclides having measurable concentrations detected by gamma spectroscopy were Cs-134 and -137.

Full-depth gross gamma scans were made within the resin beds of the prefilters, after removing the cores. Those scans were used to estimate the total integrated dose absorbed by the resins.

Estimating Radiation Doses

Gamma radiation doses were estimated for the resin samples, using gamma dose measurements obtained during full-depth, gross gamma scans within the prefilter resin beds (Ref. 2). Measurements obtained at elevations of interest were used with the method outlined in Ref. 9 to estimate the total integrated beta-gamma radiation dose for each resin sample. The estimated doses are given in Table I.

TABLE I

Estimated Radiation Doses for EPICOR-II Irradiated Resin Samples

Sample	Measured Gamma Activity at Location of Sample in Prefilter (R/min)	Gamma Radiation Dose (R)	Total Beta-Gamma Radiation Dose (rad)
PF-8#1	20	7.6×10^7	1.2×10^8
PF-8#2	11	4.7×10^7	0.7×10^8
PF-8#3	21.3	7.9×10^7	1.8×10^8
PF-20	10	4.7×10^7	0.7×10^8

Sampling

At the Test Reactor Area of INEL, each resin-filled coring tool was transferred from its cask into the Hot Cell for remote removal of resin samples. The coring tool shutter was withdrawn to expose the layers of different ion exchange media (resin). It was noted that some smearing of material from one layer into another had occurred when the shutter had been inserted during coring operations and withdrawn for sampling. That smearing of one layer into another required careful removal of the mixed surface material to expose unmixed resin near the center of the core. It was the unmixed material that was the target for collection.

The collection of resin samples was accomplished using a vacuum pump and water-filled, graduated glass column. One end of a flexible rubber tube was attached near the top of the glass column, and the other end to a vacuum pump outside the Hot Cell. A segment of rubber tubing was attached from the top of the glass column to a stainless steel tube to form a wand. With the vacuum pump running, the wand was positioned over the target resin with a master-slave manipulator in such a way that resin was drawn into the wand and thence the column. Sample sizes of 100 mL were collected. Three samples were obtained from the PF-8 core, two of strong acid cation resin (PF-8#1 and PF-8#3) and one of phenolic cation resin (PF-8#2). A single strong acid cation resin sample was obtained from PF-20. The resin samples were collected from or near those regions of

highest radionuclide loading. [This study is concerned with degradation of organic ion exchange resins; therefore, only organic resin samples were removed from the cores. No anion samples were collected from either core because of the much lower radionuclide content of the anion exchange resin (indicated by gamma scans) and accompanying low radiation dose (resulting in much less degradation than in the cation exchange resins).]

Sample Preparation

The radiation levels of the samples were of such intensity that analytical work performed on the irradiated resins would have to have been done within a hot cell environment. However, that would have made characterization and analysis of the samples very costly and time-consuming. Previous tests performed at INEL on unirradiated resins had shown that an Epicor resin could be stripped of 99% of its cations.¹⁰ Based on that information, it was decided to strip the radionuclides from the PF-8 and -20 resin samples, using a 10% hydrochloric acid solution.

As described in the previous section, samples were removed from the coring tools and drawn into separate ion exchange columns filled with distilled water. The samples were allowed to soak 24 hours in the water-filled columns. At the end of that time, the distilled water used to soak each of the samples (PF-8#1, #2, and #3, and PF-20) in the separate columns was discolored brown.

Each ion exchange column was reconfigured, and the distilled water was removed through the shutoff valve of the column and retained for gas chromatography (GC) analysis. The samples were rinsed three times each with distilled water, which was added by the pump through the tubing at the top of each column. Resin samples PF-8#1 and #3 showed restriction to flow during this initial rinse procedure. That distilled water also was retained for GC analysis and functional group tests.

A solution of 10% hydrochloric acid was pumped through each resin sample at a rate of 100 mL/min. That was continued until 55 sample volumes (the amount determined to remove 99% of the cations) or 5.5 L of acid flowed over each resin sample. Representative quantities of that acid rinse were collected and later analyzed.

The distilled water soak, distilled water rinse, and hydrochloric acid rinse greatly reduced the radionuclide content of the resins. That made it possible to remove the samples from the Hot Cell and perform the analyses in a Type II fume hood containing a high-efficiency particulate air filter on the outlet duct.

Characterization of Unirradiated and Irradiated Resins

The following analytical methods were used to characterize the unirradiated Epicor, Inc. resins (strong acid cation and phenolic cation) and samples from PF-8 (two strong acid cation samples and one phenolic cation sample) and PF-20 (strong acid cation):

- ASTM Procedures for the Physical and Chemical Properties of Particulate Ion Exchange Resins¹¹
- Infrared spectroscopy
- Gas chromatography
- Barium chloride precipitation for determination of sulfonic acid groups
- Scanning electron microscopy.

ASTM Tests. ASTM procedures were used to determine the chemical and physical conditions of the ion exchange resins. For details of those procedures see Refs. 2 and 11. Results from analysis of the irradiated EPICOR-II resins were compared with results from the unirradiated resins to determine if degradation had occurred. The following ASTM procedures^a were used for the strong acid cation and phenolic cation exchange resins:

1. The pretreatment phase of the ASTM procedure was used to convert the ion exchange resins to one standard form (usually the sodium form for cation resins). This standard form provided a baseline from which the other ASTM tests could be performed.
2. The water retention capacity test indicates the porosity of the resin. The porosity of a resin is dependent on the amount of effective cross-linking. The higher the water retention capacity, the lower the effective cross-linking. In the case of the PF-8 and -20 resins, the water retention capacity is an indication of the amount of divinylbenzene cross-linking.
3. The backwashed and settled density test was used to determine changes in effective cross-linking between new and used resins. The density is proportional to the amount of effective cross-linking in the resin.
4. The salt-splitting capacity test is designed to show the number of sulfonic acid groups contained in a cation ion exchange resin. A decrease in salt-splitting capacity would show a loss of functional sulfonic acid groups. Phenolic, carboxylic acid, and phosphonic acid functional groups also will exhibit, to some degree, salt-splitting capacity.
5. The total exchange capacity test is used to determine the exchange capacity of cation ion exchange resins that contain functional groups in addition to, or different from, sulfonic acid functional groups.

Infrared Spectroscopy. Infrared spectroscopy (IR) was used to determine if the styrene and phenolic cation resins in PF-8 and -20 were the same as the unirradiated styrene cation and phenolic cation resins obtained from Epicor, Inc. IR traces of the unirradiated resins were compared with IR traces from PF-8 and -20 resins to determine if any changes in molecular structure of the PF-8 and -20 resins had occurred because of the high internal radiation dose.

Gas Chromatography. Gas chromatography (GC) is the technique by which a liquid sample is vaporized and separated into components by means of a GC column containing a mobile phase and a stationary phase. In the case of the EPICOR-II resin samples, a 1/8-in.-OD by 6-ft-long GC column containing 0.34% tetranitrofluorenone as the mobile phase and Carboxpack C as the stationary phase was chosen for the GC analysis. Any organic products (divinylbenzene or styrene) in the original sample were concentrated by use of methylene chloride.

a. Those tests were performed in accordance with ASTM standards, and deviations are within allowable limits of those standards.

Barium Chloride Precipitation for the Determination of Sulfonic Acid Groups. It has been shown that the EPICOR-II cation resins are sulfonic acid, divinylbenzene, styrene type resins (Ref. 6). The high internal radiation dose received by those resins could cause loss of the functional groups (sulfonic acid). This loss would cause an increase in the sulfate concentration of the distilled water soak and rinse solutions from the PF-8 and -20 strong acid cation resins.

Scanning Electron Microscopy. To determine the physical conditions of the resin samples from PF-8 and -20, scanning electron microscope (SEM) photomicrographs were obtained of the resins at different magnifications. The photomicrographs allowed examination of the resins for cracks, bead breakage, and so forth.

RESULTS

Because of the age of the unirradiated resins (4 years old), they might have been expected to show some degradation. The unirradiated resins, however, showed no apparent change from previous analyses (Refs. 2 and 6). The irradiated resins from PF-8 and -20 (6 years old) also would have been expected to show some degradation due to age.^{12,13} Since the unirradiated resins showed no degradation because of age, in this study, any degradation of the irradiated resins was assumed to be from radiation damage and not from age.

ASTM Tests

Results of the tests performed on irradiated and unirradiated resins from the second sampling of PF-8 and -20 are listed in Table II. [Results from the first sampling were reported in Refs. 2 and 3.]

Infrared Spectroscopy

Figure 1 compares the IR spectrum of the PF-8#1 resin with that of the unirradiated strong acid cation resin. Figure 2 compares the IR spectrum of the PF-20 resin with that of the unirradiated strong acid cation resin. The spectra from PF-8#1 and -20 show that (a) the resins are the same as the strong acid cation resin received from Epicor, Inc. and (b) no significant changes in polymer structure are apparent in the irradiated resins from the two EPICOR-II prefilters. [The spectrum of the PF-8#3 resin was identical to that of PF-8#1.]

Figure 3 compares the IR spectrum of the PF-8#2 phenolic cation resin with that of the Epicor, Inc. supplied, unirradiated phenolic cation resin. The spectra show no differences, which indicates (a) the phenolic resin from PF-8#2 and the phenolic resin from Epicor, Inc. are the same and (b) no significant changes in polymer structure have occurred in the irradiated resins from the EPICOR-II prefilters.

Gas Chromatography

Comparing chromatograms of the PF-8#1 and PF-20 cation resins (Ref. 2) showed that no soluble organic products such as divinylbenzene or styrene were present in the soak and rinse solutions from the PF-8 and -20 strong acid cation resin samples.

Comparing the chromatogram of the PF-8#2 phenolic resin (Ref. 2) with that of the unirradiated resin showed no soluble organic products such as divinylbenzene or styrene were present in the soak and rinse solutions from the PF-8 phenolic cation resin sample, as would be expected.

TABLE II

Results of ASTM Tests on Irradiated and Unirradiated Ion Exchange Resins--Second Sampling

ASTM Test Parameter	Resin Sample					
	PF-8#1 Strong Acid Cation	PF-8#2 Phenolic Cation	PF-8#3 Strong Acid Cation	PF-20 Strong Acid Cation	Unirradiated Strong Acid Cation	Unirradiated Phenolic Cation Resin
Water retention capacity	54.40 ± 0.21%	56.55 ± 0.22%	56.70 ± 0.22%	52.00 ± 0.21%	48.20 ± 0.19%	52.50 ± 0.21%
Backwashed and settled density (g/mL)	0.73 ± 0.004	0.62 ± 0.003	0.68 ± 0.004	0.76 ± 0.004	0.80 ± 0.004	0.63 ± 0.003
Salt-splitting capacity (meq/g) ^a	4.91 ± 0.080	3.81 ± 0.080	5.01 ± 0.080	5.25 ± 0.080	5.28 ± 0.080	3.09 ± 0.080
Total exchange capacity (meq/g) ^a	5.33 ± 0.050	6.38 ± 0.070	5.50 ± 0.060	5.58 ± 0.060	5.60 ± 0.060	6.78 ± 0.070

a. Measured in milliequivalents per gram of dry resin.

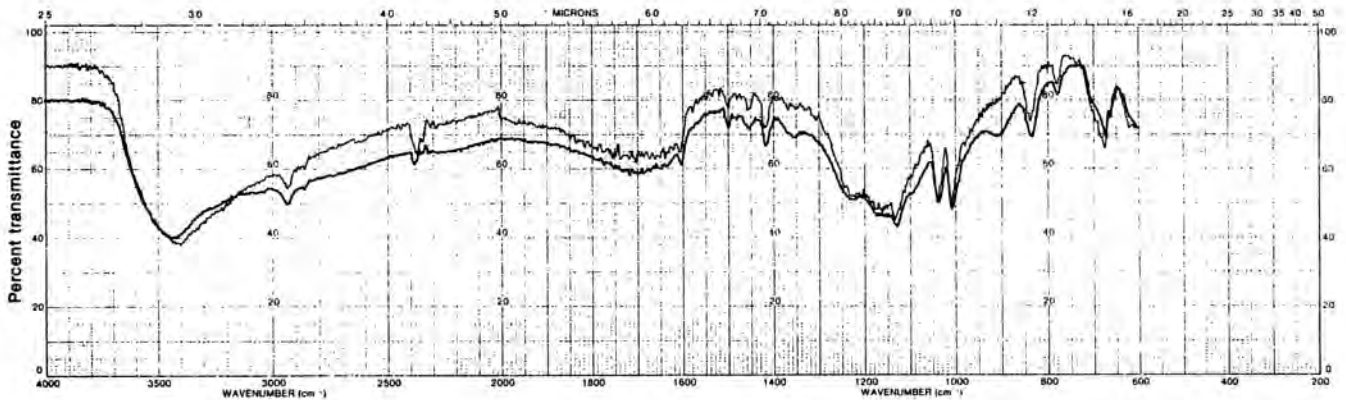


Fig. 1. Comparison of the IR Spectrum from Unirradiated, Epicor, Inc. Supplied, Strong Acid Cation Resin (Top Spectrum) With That of Sample PF-8#1 Strong Acid Cation Resin (Bottom Spectrum).

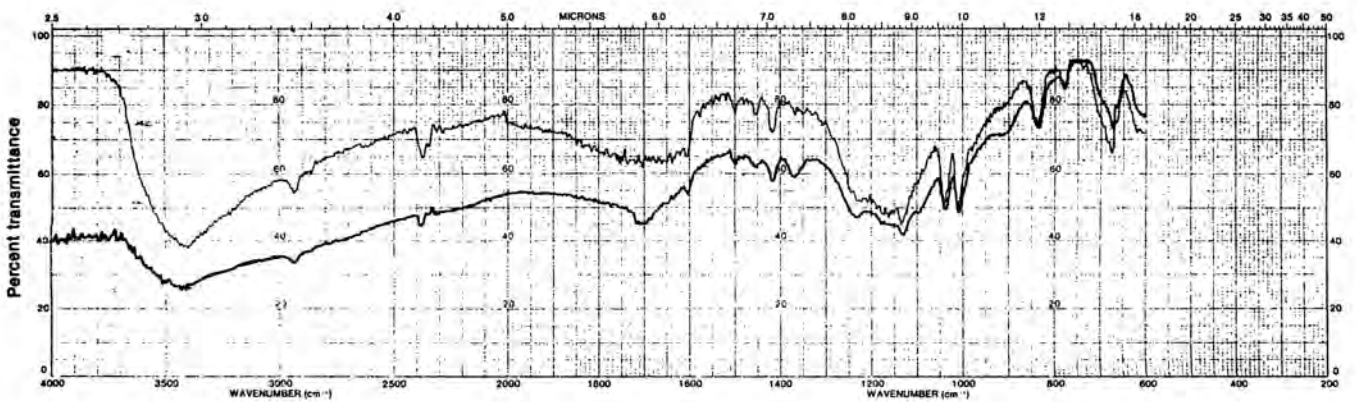


Fig. 2. Comparison of the IR Spectrum from Unirradiated, Epicor, Inc. Supplied, Strong Acid Cation Resin (Top Spectrum) With That of Sample PF-20 Strong Acid Cation Resin (Bottom Spectrum).

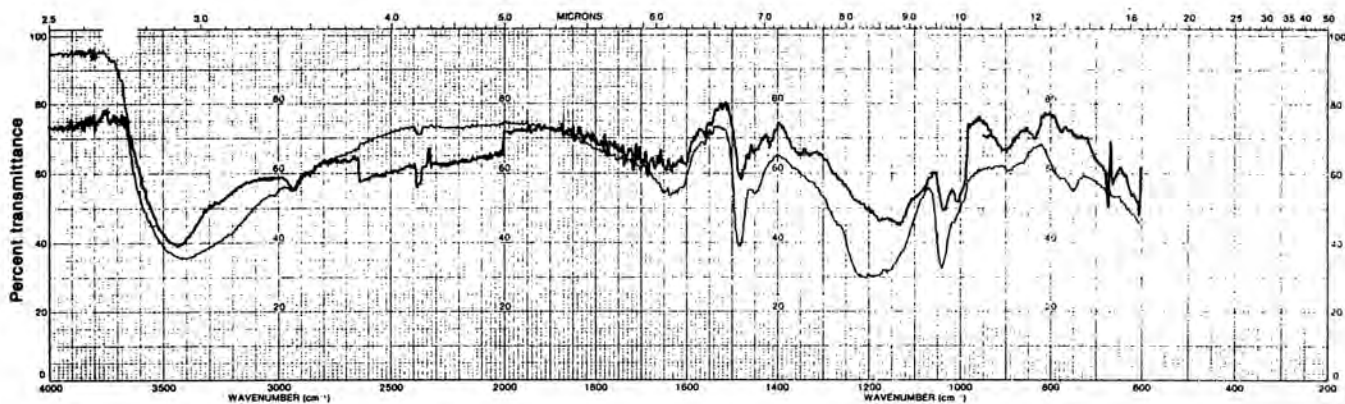


Fig. 3. Comparison of the IR Spectrum from Unirradiated, Epicor, Inc. Supplied, Phenolic Cation Resin (Top Spectrum) With That of Sample PF-8#2 Phenolic Cation Resin (Bottom Spectrum).

Barium Chloride Precipitation for Determination of Sulfonic Acid Groups

Results of the barium chloride precipitation tests showed large sulfate concentrations and low pH (less than 3) in the distilled water soak and rinse solutions for Samples PF-8#1, PF-8#3, and PF-20 strong acid cation resins. Those large sulfate concentrations indicate a loss of functional groups resulting from degradation. The phenolic resins contain no sulfonic acid groups; therefore, those samples showed no sulfate (Refs. 2 and 6).

Scanning Electron Microscopy

SEM photomicrographs of the unirradiated Epicor, Inc. supplied resins were presented in Refs. 2 and 3. Figures 4 through 7 show examples of resins from the second sampling of the irradiated EPICOR-II resins.

The SEM photomicrograph of PF-8#1 resin (Fig. 4) shows considerable damage to the resin bead. It shows

the same type of bead cracking observed during the first sampling. Photomicrographs of PF-8#3 resin (Figs. 5 and 6) show a different type of cracking. Figure 5 shows a bead which has cracked and degraded, and appears spongy. Figure 6 shows bead cracking similar to that seen in Fig. 4.

The SEM photomicrograph of PF-8#2 phenolic resin (Fig. 7) shows that particles of resin obtained from the second sampling were not as damaged physically as several of those from the first sampling (Refs. 2 and 3).

Analytical Results

Table III presents results of the various analytical tests performed on the irradiated EPICOR-II resin samples. It should be noted that results in the table are expressed in terms of differences in values obtained from tests on the irradiated EPICOR-II resins from the first and second samplings versus the values obtained from tests on the unirradiated Epicor, Inc. supplied resins.



Fig. 4. SEM Photomicrograph of EPICOR-II Strong Acid Cation Resin Bead From PF-8#1 at 100 Magnification.



Fig. 5. SEM Photomicrograph of EPICOR-II Strong Acid Cation Resin Beads From PF-8#3 at 50 Magnification.



Fig. 6. SEM Photomicrograph of Other EPICOR-II Strong Acid Cation Resin Beads From PF-8#3 at 50 Magnification.



Fig. 7. SEM Photomicrograph of EPICOR-II Phenolic Cation Resin from PF-8#2, Showing a Closeup of One Resin Particle at 50 Magnification.

TABLE III

Summary of Results From Analysis of EPICOR-II Irradiated Resin Samples^a

Analytical Technique	Resin Sample				
	Sampling	PF-8#1 Strong Acid Cation	PF-8#2 Phenolic Cation	PF-8#3 Strong Acid Cation	PF-20 Strong Acid Cation
ASTM Tests					
● Water retention capacity	1,2	Increase/Increase	Increase/Increase	No sample/Increase	Increase/Increase
● Backwashed and settled density	1,2	Decrease/Decrease	Decrease/Decrease	No sample/Decrease	Decrease/Decrease
● Salt-splitting capacity	1,2	Decrease/Decrease	Increase/Increase	No sample/Decrease	Decrease/No change
● Total exchange capacity	1,2	Decrease/Decrease	No change/Decrease	No sample/Decrease	Increase/No change
Infrared spectroscopy	1,2	No apparent changes in structure	No apparent changes in structure	No apparent changes in structure	No apparent changes in structure
Gas chromatography	1,2	No soluble products determined	No soluble products determined	No soluble products determined	No soluble products determined
BaCl ₂ precipitation for sulfonic acid groups	1,2	Sulfonic acid groups are being lost	This resin contains no sulfonic acid groups	Sulfonic acid groups are being lost	Sulfonic acid groups are being lost
Scanning electron microscopy	1	Resin bead cracking observed	Damage observed on a few particles	Resin bead cracking observed	No damage noted
	2	Resin bead cracking observed	No damage noted	Resin bead cracking observed	No damage noted
Visual observations	1	Nothing unusual observed	Contamination with PF-8#1 resin observed	Resin bead cracking observed	Nothing unusual observed
	2	Rinse and soak solutions showed a brown color	Rinse and soak solutions showed a brown color	Rinse and soak solutions showed a brown color	Rinse and soak solutions showed a brown color
Physical observations	1	Lack of flow during elution observed	Nothing unusual observed	Lack of flow during elution observed	Nothing unusual observed
	2	Lack of flow during elution observed	Nothing unusual observed	Lack of flow during elution observed	Nothing unusual observed

a. Results are expressed as differences in values observed for irradiated EPICOR-II resins versus unirradiated resins supplied by Epicor, Inc.

CONCLUSIONS

The INEL study of degradation of EPICOR-II organic ion exchange resins correlates with findings of other researchers (Refs. 12 through 17); and degradation has been identified in the EPICOR-II resins at a lower than predicted gamma radiation dose (Refs. 2 and 12). The internal radiation dose received by the organic ion exchange resins in EPICOR-II prefilters PF-8 and -20 was sufficient to initiate degradation at the time of analysis of resins from the first sampling. Degradation was continuing at the time of analysis of resins from the second sampling. The equilibrium of the polymer structure has been shifted towards polymer breakdown, as can be seen by the further change in parameters between the first and second analyses.

One important indicator of the capability of ion exchange media to retain radionuclides is the total exchange capacity. The four samples examined at INEL exhibited different reactions to radiation. PF-8#1 and PF-8#3, the strong acid cation resins with the highest radiation dose, showed reduced total exchange capacity. PF-20, the strong acid cation with the lowest radiation dose, showed no change. PF-8#2, the phenolic cation with a similar low dose, showed decreased capacity. Those results indicate that the PF-8#1 and PF-8#3 resins had reached a dose high enough to cause the exchange capacity to decrease, while the PF-8#2 phenolic cation sample went from no change at a lower dose to a decreased exchange capacity at the second sampling higher dose. The PF-20 resin had increased capacity at the first sampling and had returned to no change at the second sampling, indicating onset of degradation. Those changes agree with findings of Ref. 13.

The findings of this study can be related to commercial disposal of spent ion exchange media used in power reactors. Determining onset of degradation and, later, significant loss of exchange capacity with resultant loss of radionuclides will provide a data base useful in planning for, and regulation of, disposal of ion exchange resins.

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