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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 prefilters. Those prefilters 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 third sampling of resins from prefilters 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 and second samplings of those two prefilters.

INTRODUCTION

This paper discusses the resin degradation study conducted on organic ion exchange resins removed from two EPICOR-II prefilters 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 prefilters. Descriptions of the prefilters and the research being conducted is available (1,2,3).

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 prefilters were transported to INEL for temporary storage and preparation for disposal (4,5). Four prefilters 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 prefilters are kept in temporary storage casks outside the Hot Shop of Test Area North Building 607 at INEL.

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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 prefilters for approximately nine 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 prefilters for examination:

- 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 prefilters 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 prefilters 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). Vapor phase, liquid, and super critical fluid chromatography were used to analyze the rinse and soak solutions quantitatively for leachable organic compounds. Inductively coupled plasma-atomic emission spectroscopy 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 prefilters PF-8 and -20 in 1983, 1985, and 1989 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 and shutter, used for collecting, transporting, and storing the resin core; (b) 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 (c) vibrator tool, which drives the coring tool, casing, and shutters into the resin bed. NUREG/CR-4150 (2) further describes the coring equipment.

Gamma Scanning

Full-length isotopic gamma scans were made of each resin core from the first and second samplings to produce axial profiles 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 (2,3). Measurements obtained at elevations of interest were used with the method outlined (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	Calculated Gamma Activity at Location of Sample in Prefilter (R/min)	Total Gamma Radiation Dose (rad)	Total Beta-Gamma Radiation Dose (rad)
PF-8#1	19.4	10.9×10^7	1.5×10^8
PF-8#2	11.8	6.6×10^7	0.9×10^8
PF-8#3	19.4	10.9×10^7	1.5×10^8
PF-20	10.9	5.9×10^7	0.8×10^8

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 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 and low radiation dose indicated by gamma scans 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 examined visually.

Each ion exchange column was reconfigured, and the distilled water was removed through the shutoff valve of the column and retained for 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 and PF-20 showed restriction to flow during this initial rinse procedure and required a batch rinse with water removed by decanting. 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):

- o ASTM Procedures for the Physical and Chemical Properties of Particulate Ion Exchange Resins (11)
- o Gas, liquid, and supercritical fluid chromatography
- o Inductively coupled plasma-atomic emission spectroscopy for determination of sulfonic acid groups
- o Scanning electron microscopy.

ASTM Tests

ASTM procedures were used to determine the chemical and physical conditions of the ion exchange resins (2,11). 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.

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

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 DB5 30-m-long Durabond column with a 1.5 microfilm thickness from J. W. Scientific was chosen for the GC analysis. Any organic products in the original sample were concentrated by use of hexane.

Liquid Chromatography

High-performance liquid chromatography (HPLC) is the technique by which a liquid is separated into its components by means of liquid solid chromatography, partition chromatography, ion-exchange chromatography, or exclusion chromatography. This study relied on the technique of partition chromatography, an HPLC technique in which the solute is partitioned between two immiscible solvents, one fixed and the other mobile. The fixed phase was a HPLC ODS C-18 column from Waters Chromatography, and the mobile phase was acetonitrile. Aliquots of the hexane extracts of the soak-and-rinse solutions from PF-8 and -20 were injected into a Kratos HPLC using a UV-VISIBLE Detector, and HPLC chromatograms were obtained.

Supercritical Fluid Chromatography

Supercritical fluid chromatography (SFC) is a chromatography technique in which the mobile phase has been raised above its critical temperature and pressure. At this point, called the critical point, the mobile phase is neither a gas nor a liquid. Separations are made based on the different solubilities of analytes by changing the density of the mobile phase. This techniques lends itself well for thermally labile compounds and larger molecular weight polymeric materials which cannot be run using other chromatography techniques. Extracted liquid samples from the EPICOR-II resins were analyzed using a 10 meter SB-Phenyl-50, 100 micron ID, 0.25 micron film thickness capillary column. Carbon dioxide was used as the mobile phase and an isothermal (100^{o}C) pressure program was run starting at 100 atm and going to 375 atm at a rate of 5 atm/min.

Inductively Coupled Plasma - Atomic Emission Spectroscopy for the Determination of Sulfur (ICP-AES)

It has been shown that the EPICOR-II cation resins are sulfonic acid, divinylbenzene, styrene type resins (6). The high internal radiation dose received by those resins could cause loss of the active sulfonic acid sites. This would result in finding sulfate in the distilled water soak and rinse solution from the PF-8 and PF-20 strong acid cation resins. ICP-AES is an elemental technique and can detect sulfur present in low concentrations in aqueous solutions. This work was performed on a Perkin Elmer Plazma II ICP-AES using three analytical wave lengths for sulfur (180.731, 182.034, 182.624 nm).

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 (7 years old), they might have been expected to show some degradation. The unirradiated resins, however, showed little apparent change from previous analyses (2,3,6). The irradiated resins from PF-8 and -20 (9 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 third sampling of PF-8 and -20 are listed in Table II. [Results from the first and second samplings were previously reported (2,3)].

Gas, Liquid, and Supercritical Fluid Chromatography

The irradiated resins were soaked and then rinsed with deionized water. These water samples were then extracted with hexane in an attempt to determine if water soluble organic molecules could be leached from the irradiated resins. In all three chromatography experiments no detectable organic material was found. This would imply that the degradations products of the polystyrene divinylbenzene are not readily extractable into water. Therefore, organic decomposition products are expected to stay with the intact resin beads.

Inductively Coupled Plasma - Atomic Emission Spectroscopy

Assay of the solutions collected during resin treatment clearly demonstrate that sulfonic acid groups are being lost due to irradiation of the sulfonic acid containing polystyrene divinylbenze based resins. (See Table III). Some sulfate also was found in the PF-8#2 phenolic resin sample. This can be explained by the fact that this sample was taken next to the strong acid resin. These resin samples appeared to be still moist which would allow the sulfate to migrate slowly. Also a small amount of the strong acid cation resin was collected with the phenolic resin due to the proximity of these resins in the sampling tool.

Scanning Electron Microscopy

SEM photomicrographs of the unirradiated Epicor, Inc. supplied resins were presented in NUREG/CR reports (2,3). Figures 1 through 4 show examples of resins from the third sampling of the irradiated EPICOR-II resins.

TABLE II

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

	Resin Sample					
ASTM Test Parameter	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	56.56 ± 1.21%	52.78 ± 1.13%	56.06 ± 1.20%	48.53 ± 1.04%	41.48 ± 0.89%	45.36 ± 0.97%
Backwashed and settled density (g/mL)	0.812± 0.011	0.705± 0.009	0.829± 0.011	0.886± 0.012	0.837± 0.011	0.665± 0.009
Salt-splitting capacity (meq/g)[a]	3.78 ± 0.70	2.17 ± 0.040	3.83 ± 0.71	4.42 ± 0.82	5.21 ± 0.96	2.94 ± 0.54
Total exchange capacity (meq/g)[a]	3.44 ± 0.53	4.65 ± 0.72	3.64 ± 0.56	4.77 ± 0.74	6.28 ± 0.97	6.42 ± 0.99

a. Measured in milliequivalents per gram of dry resin.

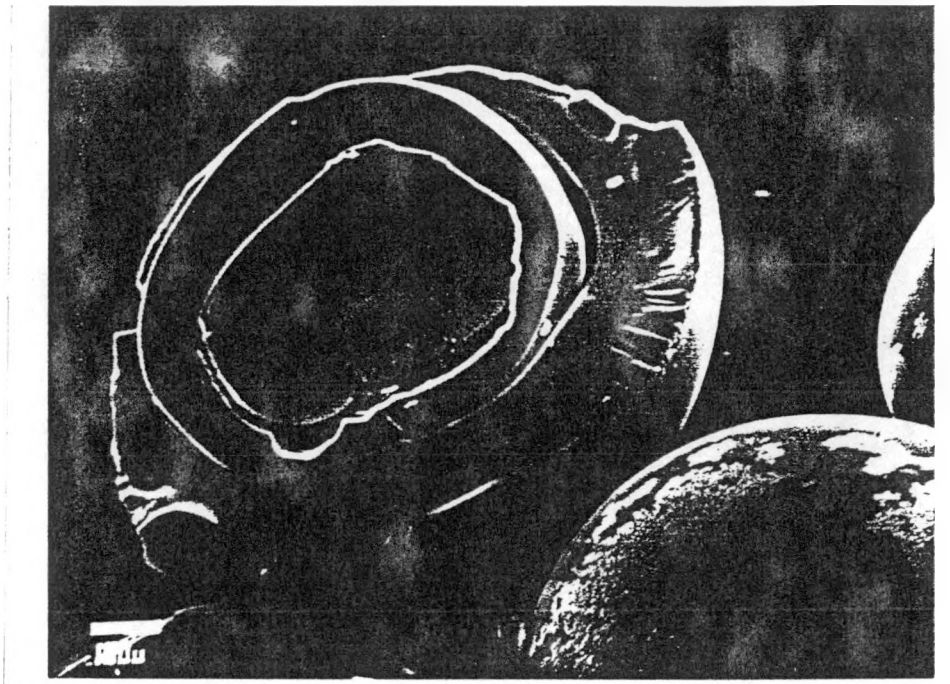

Fig. 1. SEM photomicrograph of an EPICOR-II strong acid cation resin bead from strong acid cation resin bead from PF-8#1 at 100 magnification.

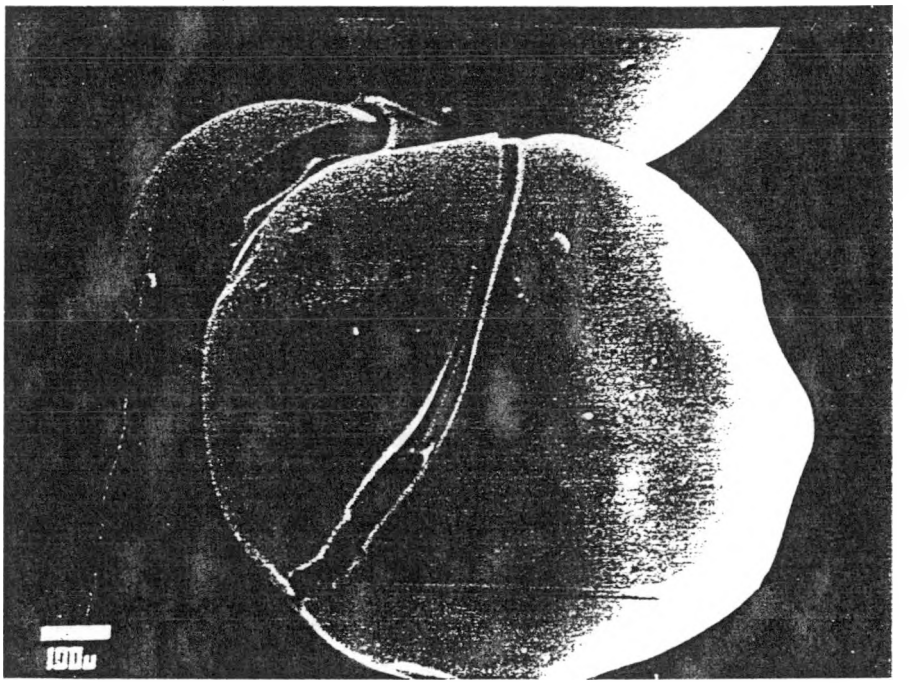

Fig. 2. SEM photomicrograph of an EPICOR-II strong acid cation resin beads from PF-8#3 at 100 magnification.

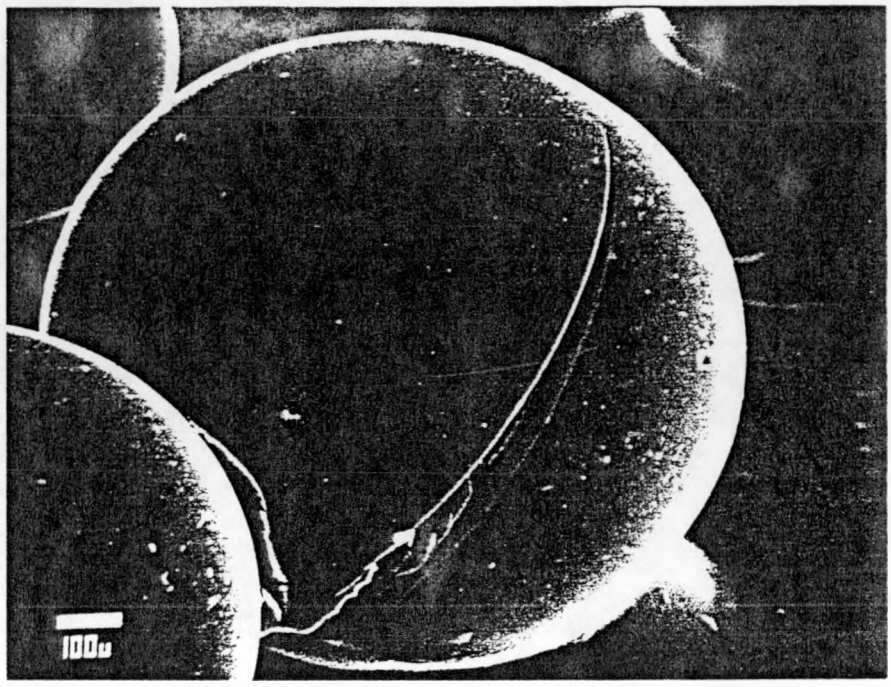

Fig. 3. SEM photomicrograph of EPICOR-II strong acid cation resin beads from PF-20 at 100 magnification.

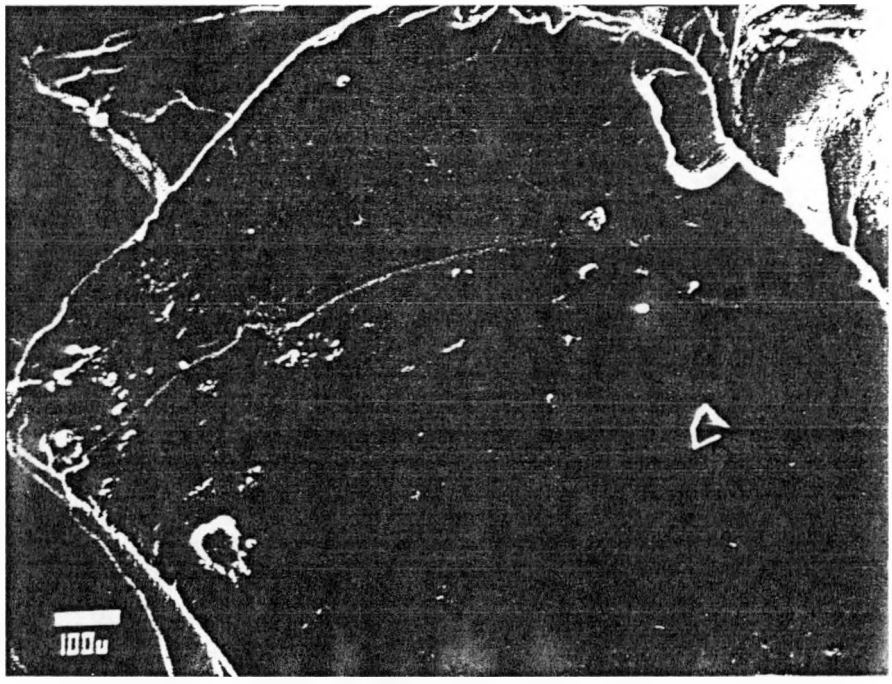

Fig. 4. SEM photomicrograph of EPICOR-II phenolic cation resin from PF-8#2, showing a closeup of one resin particle at 100 magnification.

The SEM photomicrograph of PF-8#1 resin (Fig. 1) shows considerable damage to the resin bead. It shows a concentric type of bead cracking not observed during the previous samplings. The photomicrograph of PF-8#3 resin (Fig. 2) shows a different type of cracking. Figure 2 shows two beads which have cracked. One also appears to have been deformed by surrounding beads.

The SEM photomicrograph of PF-20 resin (Fig. 3) shows a cracked bead which exhibits other damage that is partially hidden by an adjoining bead.

The SEM photomicrograph of PF-8#2 phenolic resin (Fig. 4) shows one particle obtained from the third sampling with slight cracking.

Table III. INDUCTIVELY COUPLED PLASMA - ATOMIC EMISSION SPECTROSCOPY RESULTS FOR SULFATE

Total SO4 per Resin Sample	(Milligrams SO4/100ml Sample)
PF-8#1	15
PF-8#2	4
PF-8#3	63
PF-20	37
Unirradiate Resin	0.3

Physical Observations

PF-8#1 and PF-8#3 both appeared dark orange to brown in color. The old unirradiated resin was still a light amber color. PF-20 resin samples also had a significant change in color (mostly orange). When the sampling was performed the PF-8#1 and PF-8#3 resin samples also appeared to be sticky and tenacious toward the sampling wand. In several instances the vacuum wand had to be rinsed in water to clear the vacuum tube.

Analytical Results

Table IV 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, second, and third samplings versus the values obtained from tests on the unirradiated Epicor, Inc. supplied resins.

TABLE IV

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

Analytical Technique	Sampling	Resin Sample PF-8#1 Strong Acid Cation	PF-8#2 Phenolic Cation	PF-8#3 Strong Acid Cation	PF-20 Strong Acid Cation
ASTM Tests					
o Water retention capacity	1,2,3	Incr/Incr/Incr	Incr/Incr/Incr	No sample/Incr/Incr	Incr/Incr/Incr
o Backwashed and settled density	1,2,3	Decr/Decr/Decr	Decr/Decr/Decr	No sample/Decr/Incr	Decr/Decr/Incr
o Salt-splitting capacity	1,2,3	Decr/Decr/Decr	Incr/Incr/Decr	No sample/Decr/Decr	Decr/No change/Decr
o Total exchange capacity	1,2,3	Decr/Decr/Decr	No change/Decr/Decr	No sample/Decr/Decr	Incr/No change/Decr
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, Liquid and Supercritical Fluid Chromatography	1,2,3	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	Sulfonic acid groups are being lost	Sulfonic acid groups are being
Induction Coupled Plasma - Atomic Emission Spectroscopy	3	Sulfonic acid groups are being lost	Some sulfonic acid groups found	Sulfonic acid groups are being lost	Sulfonic acid groups are being lost
Scanning electron microscopy Observations	1	Resin bead cracking	Damage on a few particles	Resin bead cracking	No damage noted
	2	Resin bead cracking	No damage noted	Resin bead cracking	No damage noted
	3	Resin bead cracking	Damage on one particle	Resin bead cracking	Damage to one bead

TABLE IV (cont.)

Analytical Technique	Sampling	Resin Sample: PF-8#1 Strong Acid Cation	PF-8#2 Phenolic Cation	PF-8#3 Strong Acid Cation	PF-20 Strong Acid Cation
Visual observations	1	Nothing unusual	Contamination with PF-8#1 resin	Resin bead cracking	Nothing unusual
	2	Soak and rinse solutions were a brown color	Soak and rinse solutions were a brown color	Soak and rinse solutions were a brown color	Soak and rinse solutions were a brown color
	3	Soak and rinse solutions were light brown color with sediment	Soak solution contained light brown sediment	Soak and rinse solutions were light brown color with sediment	Soak and rinse solutions were light brown color with sediment
Physical observations	1	Lack of flow during elution	Nothing unusual	Lack of flow during elution	Nothing unusual
	2	Lack of flow during elution	Nothing unusual	Lack of flow during elution	Nothing unusual
	3	Lack of flow during elution	Nothing unusual	Lack of flow during elution	Lack of flow during elution

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 (12-17); and degradation has been identified in the EPICOR-II resins at a lower than predicted gamma radiation dose (2,3,12). 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 and increased noticeably by the third 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, second, and third 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 further reduced total exchange capacity. PF-20, the strong acid cation with the lowest radiation dose, showed a decrease from no change. PF-8#2, the phenolic cation with a similar low dose, showed further decreased capacity. Those results indicate that the PF-8#1 and PF-8#3 resins and the PF-8#2 phenolic cation sample had reached a dose high enough to cause the exchange capacity to rapidly decrease. The PF-20 resin had moderately decreased capacity at the third sampling indicating further progress of degradation. Those changes agree with other findings (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 provides a data base useful in planning for, and regulation of, disposal of ion exchange resins.

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