Ion Exchange Media for Reduction of Liquid Radwaste in Commercial Power Plants - 8194

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ABSTRACT

Ion exchange resins currently make up as much as one-half of all radioactive waste generated by commercial nuclear power plants. A major challenge is reduction of the quantity of ion exchange media requiring disposal. Although the amount of spent ion exchange resins disposed has decreased year after year, a new urgency has arisen with the pending closure of a major disposal site in 2008. This paper explores whether ion exchange resins also can be used to potentially reduce radioactive liquid waste volumes and / or limit them to Class A wastes only.

Source term reduction and minimization of manpower exposure to radioactivity are other important goals. Specialty ion exchange products may help to achieve source term reduction of certain radionuclides. Some established operations, data, and process concepts are presented to address these critical issues encountered in liquid radwaste management.

INTRODUCTION

Ion exchange media are used in many applications throughout the nuclear power industry. These media include synthetic organic ion exchange resins and inorganic media comprised of both natural and synthetic zeolites. Moreover, the ion exchange media used encompass a variety of physical forms including powders, beads, granules, and composites. Other physical characteristics of the media such as porosity, surface area, particle size, permeability, and stability play a role in the utility of these media in the power industry.

Control of radionuclides is an important goal throughout the nuclear industry. Ion exchange media constitute a primary means of exercising that control. In boiling water reactor (BWR) plants these media are employed in condensate polishers, reactor water cleanup (RWCU), fuel pool, and radwaste applications. Some of the most critical radionuclides in BWR's are isotopes of cobalt, copper, and iron. In pressurized water reactor (PWR) plants, ion exchange media are utilized in condensate polishers, blowdown demineralizers, chemical volume control systems (CVCS), and radwaste applications. For PWR's crictical radionuclides include isotopes of cesium, cobalt, antimony, and iron.

Ion exchange resins are ideally suited to remove soluble species including high activity, low concentration radionuclides. Synthetic organic resins (strongly basic anion, strongly acidic

cation, and mixed beds thereof) are widely used in many of the applications listed above. Specialty resins, including inorganic exchangers, are also frequently employed to remove specific radionuclides (e.g., Cs-134, Cs-137, Co-58, Co-60, Sb-125, Fe-55) with high specificity from liquid radwaste.

Overall reduction / elimination of troublesome radionuclides in nuclear plants has been quite successful. In recent decades internal manpower exposure to radiation, activity release to the environment, and waste volumes requiring disposal all have been reduced. Nevertheless, efforts continue to minimize radionuclide generation (source term reduction), post-treatment (liquid radwaste operations), and final release (waste disposal), all of which remain priority targets in nuclear plants. Since ion exchange media play such a large role both in treating radionuclides and generating radioactive waste requiring disposal, further radionuclide reduction and control are inevitably linked to the ion exchangers used in nuclear plants.

Five specific applications of ion exchange media are discussed below. In each case, the ion exchange media are specifically selected to address an ongoing radionuclide related problem / issue in the nuclear utility industry.

PLANT EXPERIENCE / PROCESS CONCEPTS

Iron Transport Reduction
Iron transport reduction is a priority for many nuclear power plants. Corrosion product deposition is of particular concern in the steam generator. Iron and similar metal contaminants foul the internal surfaces of the steam generator. This fouling, in turn, inhibits heat transfer, reduces steam pressure, and creates an environment conducive to further localized corrosion.

A common strategy to minimize feedwater corrosion transport in PWR plants involves elevating the secondary side pH. However, conventional H^+/OH^- operation of condensate polishers limits the ability to elevate pH. Industry experience indicates that the adding dissolved oxygen to the condensate reduces feedwater iron levels. Fellers and Stevens found that such addition alters the oxidation states of iron which ultimately inhibit the diffusion of iron. [1] Even with dissolved oxygen addition, feedwater iron levels undergo seasonal variations attributable to changes in oxygen solubility with temperature.

Personnel at Dominion Energy Millstone Station[a] began investigating amine form operation of the plants condensate polishers in 2002 in an effort to further reduce iron transport. Specifically, the strongly acidic cation resin component of the condensate polisher mixed beds was converted from the hydrogen to the ethanolamine (ETA) form. Following successful laboratory tests, the first amine form condensate polisher was placed in service in March 2004. This polisher was removed from service after only 42 hours of operation due to rising steam generator chlorides. Eventually the source of chloride contamination was traced to the caustic (NaOH) used to generate the strongly basic anion resin in this polisher.

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[a] Dominion Millstone Nuclear Power Station consists of two operating PWR units: an 895 MWe Unit 2 and an 1154 MWe Unit 3. The Millstone site is located in Waterford, Connecticut and utilizes seawater cooling from Long Island Sound.
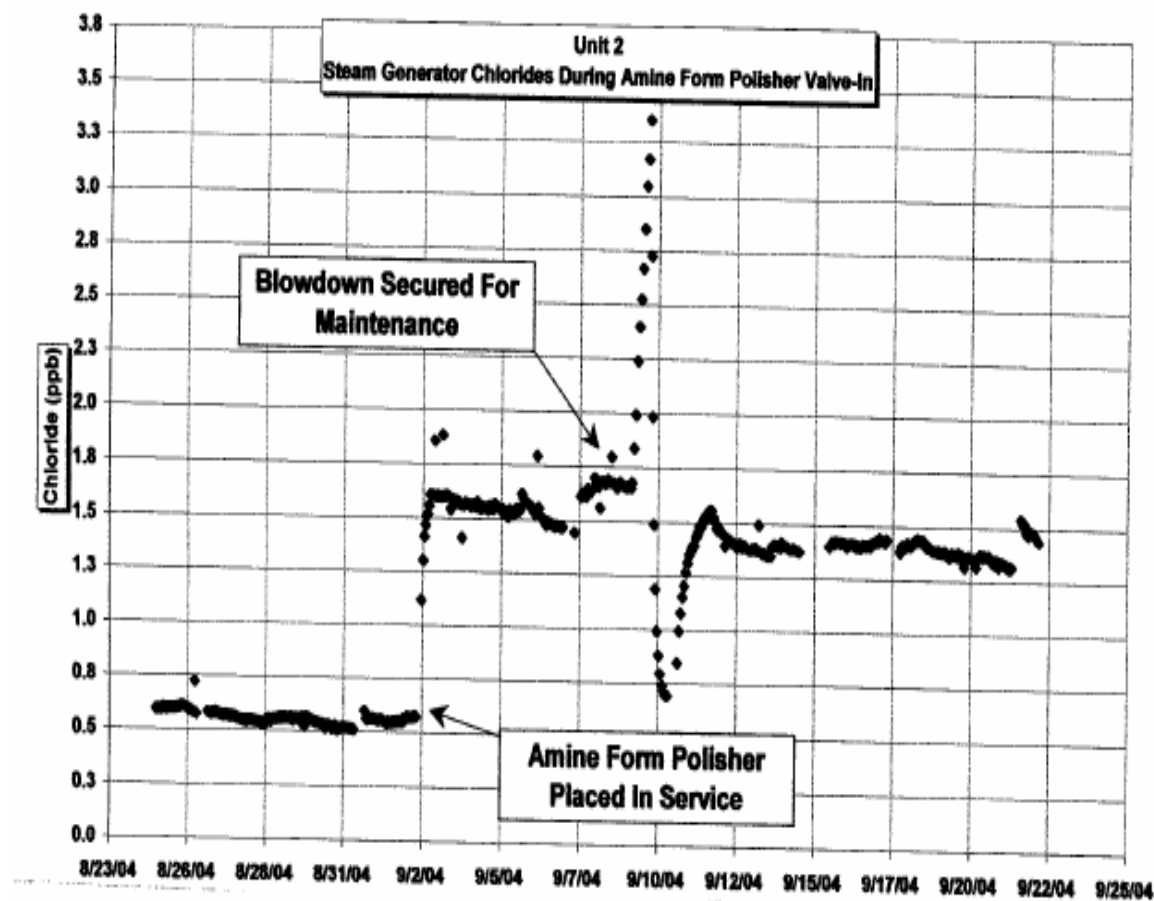
Figure 1. Steam Generator Chorides During Initial Amine Polisher Operation. [2]

At the behest of Millstone personnel, Graver Technologies developed a new high purity, ultra low chloride strongly basic anion resin, Gravex GR 1-9 Ultra, for amine form operation. The key difference between standard low chloride grade anion exchange resins and the GR 1-9 Ultra resin is residual chloride content: approximately <0.1% of the anion exchange sites for the former and <0.015% of the sites for the latter.

The first bed of GR 1-9 Ultra resin was installed a single condensate polisher vessel in Unit 2 at Millstone in September 2004, along with strongly acidic cation resin previously converted to the ETA form. Initially, steam generator chlorides increased from 0.59 µg/L to 1.4 µg/L while chloride in the effluent from this polisher was back calculated as 28 ng/L. After three months of operation, the ETA concentration in the feedwater was increased from ~1.25 g/L to 2.75 g/L. With the increased ETA, iron concentrations in the feedwater decreased from ~3.0 µg/L to ~2.0 µg/L. This mixed bed polisher remained in service for 217 days while treating >3.3 billion liters of condensate without any increase in differential pressure or decrease in flow across the bed. During a refueling outage in April 2005, this mixed bed was removed from service, mechanically cleaned, and reinstalled in the vessel without regeneration of either component. As of January 1,

2008, this GR 1-9 Ultra has been in service for >40 months (>1215 days) and has processed almost 19 billion liters of condensate. The calculated level of chloride in the effluent from this polisher is currently <10 ng/L.

The success of the first installation of the ultra low chloride anion has prompted installation of additional charges of the GR 1-9 Ultra in vessels in both Units 2 and 3 at Millstone. A total of five different lots of the GR 1-9 Ultra have been put into service at Millstone in nine separate vessels as of November 2007.   Table I summarizes the installation and service history of these lots and installed vessels. Six of the vessels remain in service currently, while three have been returned to H / OH service due to "elevated" effluent chloride levels. One charge was removed from service when the bed was contaminated during liner repairs on the vessel.

As of January 1, 2008 more than 65.6 billion liters of condensate has been processed through the ultra low chloride anion vessels with a cumulative service time of 4,235 days. Chloride concentrations in the steam generator of Units 2 have ranged between 1.2 and 1.4 µg/L throughout Cycle 18 with the amine form polishers. These values comply with the maximum 1.6 µg/L imposed by the current Condensate Polisher Index (CPI) limit. This constraint was lifted as of January 1, 2008 which, in turn, will allow installation of additional beds of GR 1-9 Ultra.

Table I. Installation and Service History for GR 1-9 Ultra Ultra Low Chloride Anion Resin

Lot Number	Installation Date	Installation Location	Service Time[b]	Throughput	Initial Effluent Chloride
			(days)	(liters X 10^9)	(ng/L)
GR-2164	09/02/04	Unit 2	1215	18.8	28[c]
GR-2374	04/07/05	Unit 3	482	7.5	33
GR-2579	01/11/06	Unit 2	719	11.1	25[c]
	01/21/06	Unit 2	114	0.5	25[d]
GR-2668	03/29/06	Unit 3	531	8.2	22
	03/30/06	Unit 3	171	2.6	27
	04/14/06	Unit 3	626	9.7	22
	05/24/07	Unit 2	221	3.4	16
GR-3100	08/09/07	Unit 2	144	2.2	19
	12/20/07	Unit 3	12	0.2	20

[b] Service time as of January 1, 2008.
[c] Current effluent chloride <10 ng/L.
[c] Current effluent chloride <10 ng/L.
[d] Anion resin contaminated during vessel repair. Effluent chloride 15 ng/L at removal.

Implementation of ultra low chloride anion resin and amine form cation resin operation allowed further elevation of secondary plant ETA levels at Millstone. For Unit 2, elevating the dissolved oxygen content in the condensate to ~12 µg/L through air injection reduced total corrosion product transport to the steam generators by almost 40%. Increasing the ETA level from ~1.25 g/L to 2.75 g/L resulted in a further reduction of final feedwater iron levels to historic lows. Average final feedwater iron concentrations continue to decline, averaging ~1.8 µg/L in June 2007 and ~1.6 µg/L in November 2007. Figure 2 summarizes the history of feedwater iron levels in Unit 2 from Cycle 14 through Cycle 17 (April 2000 through August 2005).
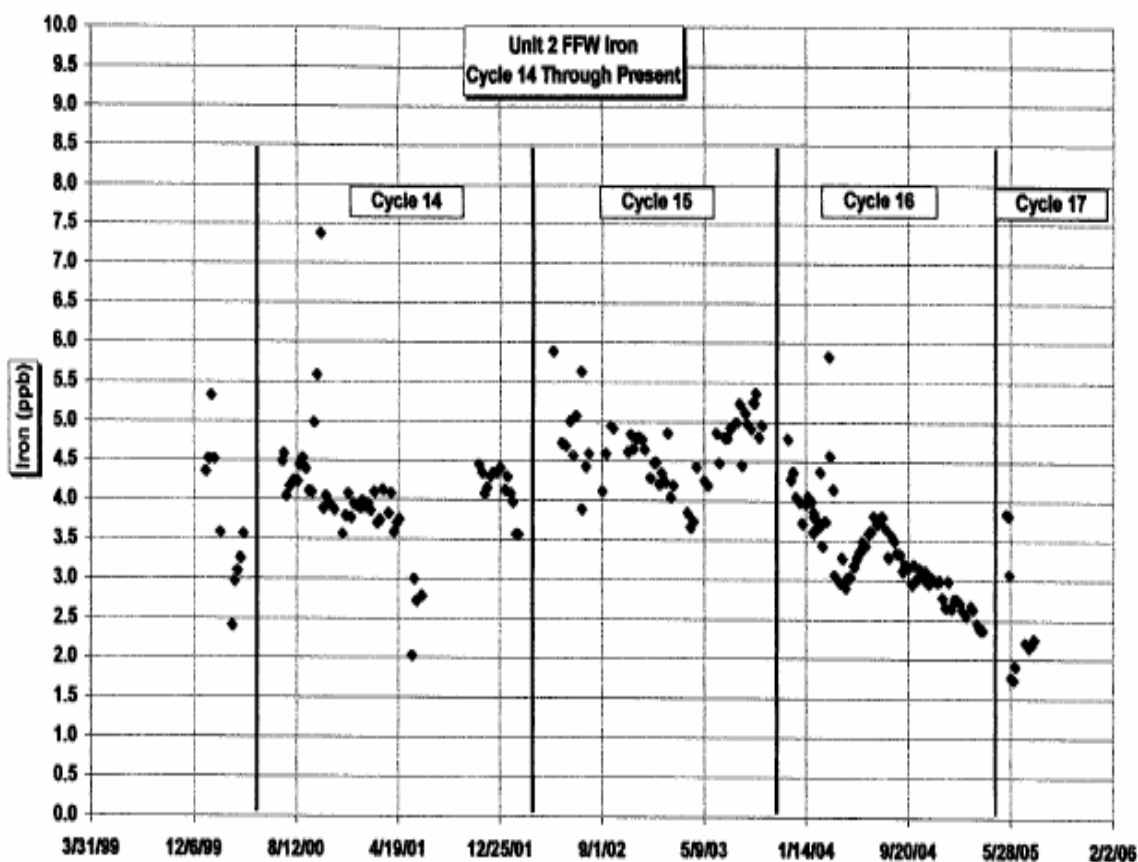

Figure 2. Unit 2 Feedwater Iron as a Function of Ethanolamine Concentration. [2]

Air injection is currently no used at Millstone Unit 3; consequently feedwater iron levels are inherently higher for that unit.   Similarly to what occurred in Unit 2, feedwater water iron levels have decreased as ETA concentration increased. Average final feedwater iron concentrations in Unit 3 have declined, averaging ~2.4 µg/L in June 2007 and ~2.0 µg/L in November 2007. Figure 3 summarizes the history of feedwater ETA and iron levels in Unit 3 from Cycle 7 through Cycle 10 (April 1999 to August 2005).
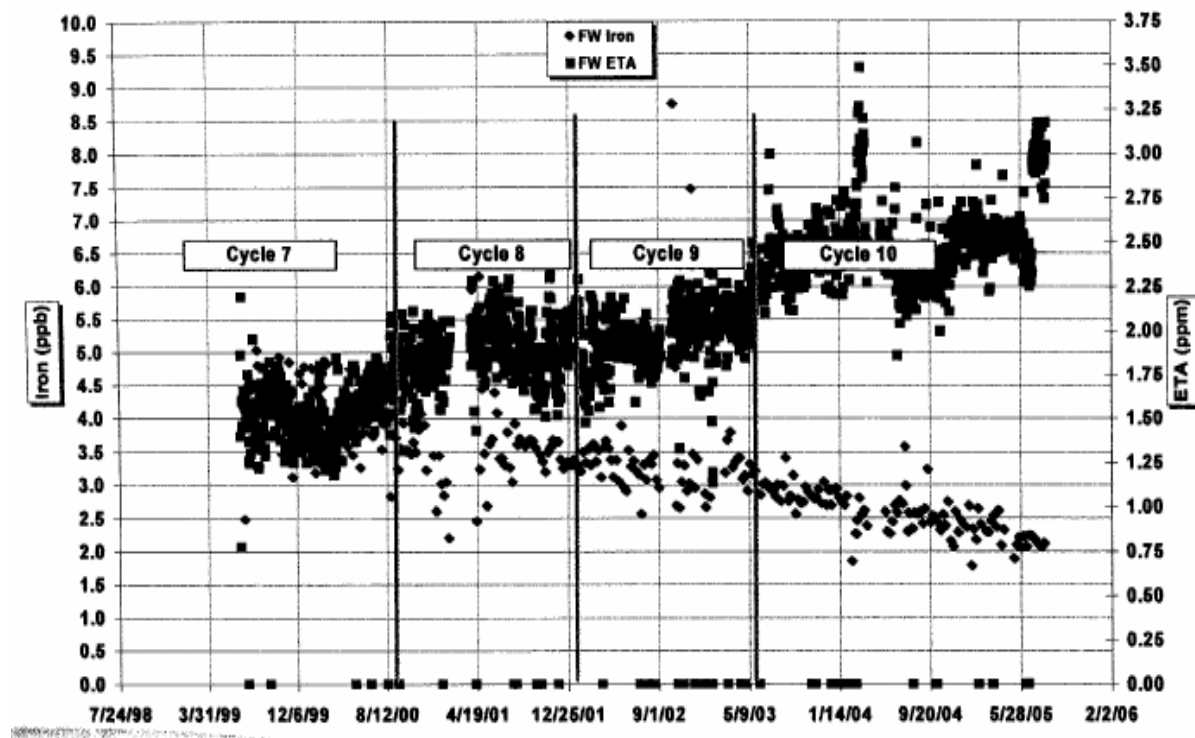
Figure 3. Unit 3 Feedwater Iron as a Function of Ethanolamine Concentration. [2]

Table II summarizes total corrosion product transport to the steam generators at Millstone as a function of unit and cycle. The decline in corrosion product transport in both units is quite dramatic in recent years.

Table II. Corrosion Product Transport to Steam Generators at Dominion Millstone Station

	Corrosion Product		Total Iron (kg)	
	Source		Unit 2	Unit 3
Cycle Number				
8				280.0
9				243.7
10				207.7
11				184.1
12				(165.3)
13				
14				
15			151.8	
16			106.8	
17			77.3	
18			(59.2)	

Antimony Selective Media
Other than cesium and cobalt, the most troublesome radionuclide in liquid radwaste of PWR's is antimony (particularly Sb-125). Unlike the cesium and cobalt, soluble antimony in oxidative conditions common to PWR's exists primarily as the anionic $Sb(OH)_6^-$ species as shown in a Pourbaix diagram [5]. Consequently, strongly basic anion resins remove soluble antimony from radwaste. Unfortunately, these resins also slough antimony unexpectedly under certain conditions such as high borate loads. A variety of metal oxides remove antimony without suffering from the sloughage issue. [6-11]

Over the last three years Graver Technologies commercialized a granular composite metal oxide media with good selectivity for radioantimony. Slip-stream testing on standard liquid radwaste containing significant radioantimony species was performed at two PWR nuclear plants. These trials yielded removal of antimony below the minimum detectable activity (MDA) level over extended time periods even with significant levels of borate present. Decontamination factors (DF's) ranged upwards from 170 based on influent radioantimony activities of greater than 4.0 E +02 Bq/L.

As a consequence of this success, EnergySolutions installed the Gravex GX187 antimony selective media in relatively shallow layered beds over existing mixed beds in their ALPS pressure vessels in the liquid radwaste processing systems at the PWR plant where the slip-stream trial was performed. The initial installation involved 0.14 m^3 (5 ft^3) of GX187 placed in service in August 2005. Both floor drain and reactor hold-up tank radwaste were processed batchwise through this media for roughly one year. Influent activity of Sb-125 ranged from 1.0 E+02 to 3.0 E+02 Bq/L. Antimony activity in the effluent from the media remained below MDA levels through the fall 2005 refueling outage and until more than 1.5 million liters (>0.4 million gallons) of radwaste were processed. Over the length of its service, the GX187 processed more than 2.1 million liters of liquid radwaste. During this time the influent Sb-125 activity averaged 3.2 E+02 Bq/L while effluent activity averaged 2.9 E+00 Bq/L which translates to an average DF of 111.

Graver Technologies subsequently developed a second generation antimony selective media, Gravex GX194, with improved physical stability. The GX194 media was installed in two additional PWR plants in early 2006. Again, the media was utilized in layered beds in ALPS systems operated by EnergySolutions. In both plants, the GX194 processed more than 1.3 million liters of radwaste batchwise during 2006. Influent Sb-125 activities in one plant ranged from 3.2 E+01 Bq/L to 2.8 Bq/L E+0 with the GX194 generating MDA quality effluent 80% of the time. At the other plant the average influent Sb-125 activity was 5.8 E+03 Bq/L while the average effluent activity was 1.5 E+02 Bq/L which translate to a DF of 39. Both plants have installed second charges of GX194 with a deeper (0.28 m^3 {10 ft^3}) layer in the ALPS vessel.

BWR Phase Separators
Some degree of substrate corrosion occurs in both BWR and PWR nuclear reactors. These corrosion products, typically iron oxides and hydroxyoxides in suspension, circulate through liquid streams in these plants. Condensate polishers remove some of the charged iron species via ion exchange. Typically, in BWR's residual iron oxides and hydroxyoxides are concentrated in the phase separators. The largest, heaviest particles settle out by gravity while finer, lighter

suspended solids are removed by precoat filtration. Gravity settling is highly time dependent, can result in high dose rates to plant personnel, and is easily disturbed when additional liquid waste is transferred into the phase separators.

Settling problems can be mitigated by the addition of extremely finely divided ion exchange resins. These extremely fine particles have massive surface areas combined with rapid kinetics. In this application, the ion exchange resins act as charged solid coagulants that assist in settling iron oxides. By contrast, typical organic coagulants do not provide the same benefit in this application. Extremely fine powders of both strongly basic anion resins (Powdex® PAO-XF) in the hydroxide form and strongly acidic cation resins (Powdex® PCH-XF) in the hydrogen form are used in phase separator applications. The two resin components are added to the phase separator sequentially to coagulate or flock the fine iron particles in suspension..

EnergySolutions and Graver Technologies collaborated to develop a Suspended Solids Concentration (SSC) technology. This approach combines the use of the extremely finely divided ion exchange resins described above combined with a tubular crossflow filtration membrane. Inorganic sintered membranes coated with titanium dioxide with uniform pores of ca. 0.1 µm deliver high fluxes even at elevated temperatures and operating flow rates of 1500 L/min. In addition, they are acid resistant which allows dissolution of any iron hydroxide fouling with dilute acid. The crossflow filtration membrane provides finer filtration and complete capture of suspended solids without sacrificing product recovery rates or high fluxes. Addition of the extremely fine ion exchange resins (Powdex® PAO-XF and Powdex® PCH-XF) add the capability of removal of dissolved ionic species as well. Very low volumes of these resins are required and the particles occupy the interstitial voids within the iron oxide sludge. Thus, there is almost no additional waste volume resulting from the use of the powdered resins. Using a simulated BWR phase separator waste stream, Tim Milner of EnergySolutions demonstrated volume reduction by a factor of 5 with a corresponding increase in solids loading as well as a clear permeate with high conductivity. [12]

Colloid Removal
During refueling outages at nuclear power plants, initial reducing conditions favor release of both oxides and metals deposited on surfaces; subsequent oxidative conditions promote formation and migration of metal oxides into the coolant. These oxides subsequently are complexed with radionuclides such as Fe-55 and Co-58. These reactions, in turn, result in so-called crud bursts of both colloidal particles and soluble ions. Colloidal metal oxides take on various physical forms such as flocked agglomerations, crystalline needles, or amorphous masses.

Colloids are defined as small particles between 1.0 nm and 5 µm in size. The largest colloidal particles are similar in size to finer suspended solids. Filtration will remove colloidal particles as long as the porosity of the filter is fine enough to operate effectively. For instance, both 0.1 µm absolute filters and even 1.0 µm absolute filters will remove larger colloidal particles. Most of the colloidal particles formed in a crud burst are smaller than 0.45 µm and thereby not efficiently removed by filtration.

Highly porous macroporous strongly basic anion exchange resins have been employed to remove natural colloids present in water supplies for many years. [13]   These colloids are combinations of aluminum, iron, silica, hydrous oxides, and organic matter found in surface waters. Recently, these highly macroporous strongly basic resins have been used to remove the metal oxide colloids present in nuclear plants [14, 15]   Smaller colloids can be trapped within the large pores of the macroporous bead structure while larger colloids as well as those of irregular shape can be adsorbed onto the surface of the beads. In addition, the largest colloidal particles can collect on top the resin bed via a filtration process.

Typically, these highly porous macroporous anion resins have been layered on top of existing resin beds (lithiated mixed bed or lithiated cation and mixed bed). Initially, they were used only during startup and refueling outages. Currently, many nuclear plants [16], including Constellation Ginna, Exelon Braidwood, First Energy Beaver Valley, FPL Seabrook, Luminant Comanche Peak, South Texas Project, TVA Sequoyah and TVA Watts Bar, are utilizing these highly porous macroporous anion resins during normal operation as well. The highly porous macroporous anion resins have also been installed as a layer over mixed beds in spent fuel demineralizers and in radwaste systems.

Physical stability has been an Achilles heel for highly porous macroporous anion resins since their inception, These resins suffer from very low crush strengths and literally can be pulverized with finger pressure. In the past, efforts to improve physical strength have inevitably reduced pore sizes. Currently, Graver Technologies is revisiting this issue. Initial laboratory testing of candidate resins has identified several promising candidates which will be submitted to an interested nuclear plant organization for evaluation using colloidal metal oxides found in their plant. At this point it is unclear exactly what minimum pore size will allow effective removal of colloidal metals. Figure 4 is a comparison of scanning electron microscope (SEM) images of two highly porous macroporous anion resins. Table III illustrates key properties of a commercial resin used in this application as well as two candidate resins.

Even the most porous macroporous strongly acidic cation exchange resins generally are less effective at removing colloids than the highly porous anion resins discussed above. This ineffectiveness may be attributable to the reducing cation environment and / or the smaller inherent pore size. The macroporous strongly acidic resins do offer improved kinetics and desirable selectivity for the common soluble cationic radionuclides (Co-58, Co-60, Cs-134, Cs-137).
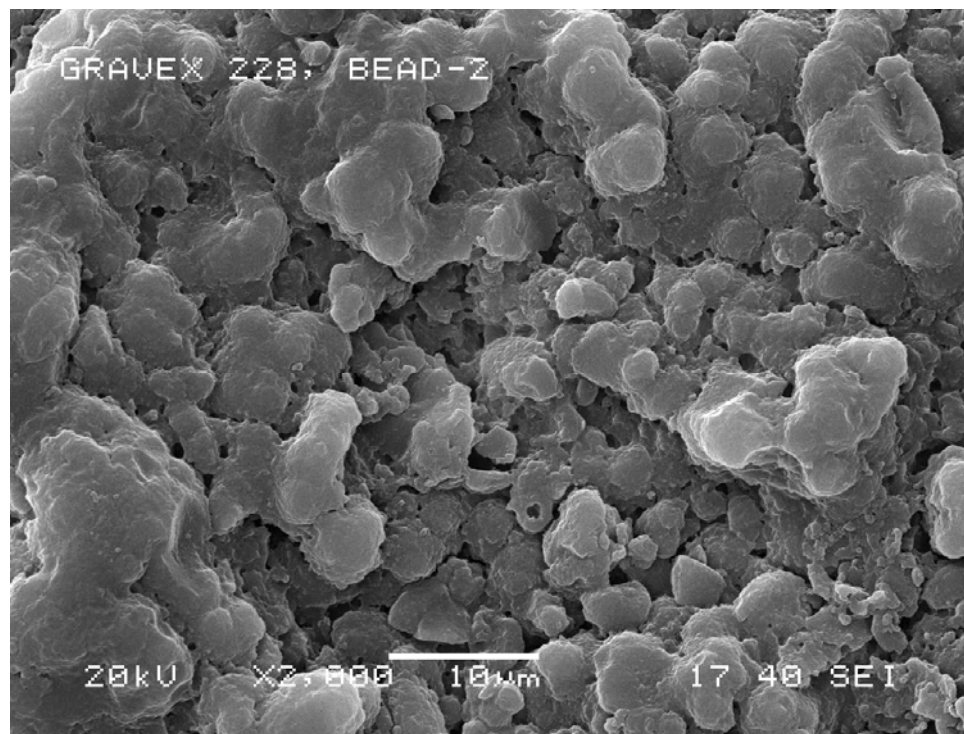
Fig. 4. SEM images of single beads of two highly porous macroporous anion resins.

Table II. Key Properties Comparison of Highly Porous Macroporous Strongly Basic Anion Resins

Resin			GX182	GX228	GX229
Ionic Form			Hydroxide	Hydroxide	Hydroxide
Property					
Exchange Capacity	(meq/mL)		0.59	0.59	1.1
Water Content	(%)		80	67	69
Whole Bead Content	(%)		95	90	97
Friability	(g/bead)		25	58	144
Surface Area	(m2/g)		4.4	39.1	44.5
Average Pore Diameter	(μm)		1.16	0.076	0.050
Porosity	(%)		69.7	69.9	59.4

Class A Waste

The Nuclear Regulatory Commission (NRC) classifies radioactive waste from commercial nuclear power plants based the concentration of long-lived radionuclides and on the concentration of short-lived radionuclides. [17]   For near surface disposal, the NRC has designated three Classes (A, B, and C) of increasing hazard.   Table IV summarizes the concentrations of non-transuranic radioisotopes that mandate either a Class B or a Class C designation.

Many nuclear utilities ship radioactive waste such as spent ion exchange resins (beads and powders) and used filters to the EnergySolutions Barnwell site for burial. Most of the spent resin meets the Class A limitations, though approximately 285 m^3 (10000 ft^3) per year fall in the Class B / C designation.   These Class B / C resins typically are used either in the reactor fuel pool (RFP), reactor water cleanup (RWCU), chemical volume control system (CVCS), or lithiated mixed beds. Elevated concentrations of the radioisotopes Cs-137 and Sr-90 push these resins into the Class B / C.

Beginning July 1, 2008 the Barnwell site will be closed to disposal of Class B / C waste except from generators within the Atlantic Compact (South Carolina, New Jersey, and Connecticut). Other utilities will be forced to store any Class B / C waste on site or change their mode of operation to eliminate generation of Class B / C waste.

One option to eliminate Class B / C waste would be to reduce the exchange capacity of a given resin or given bed. Current ion exchange resins used in the nuclear industry are fully functionalized throughout every resin particle, regardless of whether the particles are beads or powders. However, the technology exists to functionalize resin beads only on the surface of the bead. Both the pellicular ion exchange resins used in chromatographic applications and the so-called shallow shell resins [18] used in water softening applications employ this technology. Graver Technologies has developed samples of strongly acidic cation bead resin with volumetric exchange capacities below 0.2 meq/mL. For powdered resins, Graver has developed a proprietary technology to generate small composite particles that contain limited quantities of

active ingredient. This technology could be utilized to combine limited ion exchange capacity within an inert matrix.

Table IV. Radioisotope Concentrations for Class B and Class C Waste.

	Radionuclide		Class B Concentration (Ci/m^3)	Class C Concentration (Ci/m^3)
Long-Lived Species				
	C-14			0.8 – 8.
	Tc-99			0.3 – 3.
	I-129			0.008 – 0.08
Short-Lived Species				
	Ni-63[d]		3.5 – 70.	70.0 – 700.
	Sr-90[e]		0.04 – 150.	150. – 7000.
	Cs-137[f]		1. – 44.	44. – 4600.

SUMMARY

Four of the five applications for removal or reduction of radionuclides from nuclear plant operations have been fully demonstrated already. Millstone Station continues to utilize the ultra low chloride resin, Gravex GR 1-9 Ultra, to limit chloride leakage from condensate polishers, which, in turn, allows elevation of ETA and the corresponding reduction of iron transport. Several nuclear plants, including D.C.Cook, Palisades, and Sequoyah, currently utilize the granular media, Gravex GX194, to remove radioantimony from liquid radwaste. A southern BWR plant has utilized the extra fine ion exchange powders to facilitate flocculation and settling of iron in their Phase Separators for a number of years. A number of nuclear plants have employed highly porous macroporous anion resins to remove colloidal iron and cobalt metal species during various operations in recent years. The combination of finely divided ion exchange resins and crossflow filtration has been successfully demonstrated in the laboratory for removal of iron in Phase Separators. Although the reduced capacity cation resins for Class A waste restriction have been synthesized and sampled, they have not yet been tested in a nuclear plant.

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[d] Maximum concentration of Ni-63 for Class A waste classification = 5.8 X 10^{-5} g/L
[e] Maximum concentration of Sr-90 for Class A waste classification = 2.8 X 10^{-6} g/L
[f] Maximum concentration of Cs-137 for Class A waste classification = 1.2 X 10^{-5} g/L

ACKNOWLEDGEMENTS

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