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Study on immobilization of the spent ion exchange resins of Tehran research reactor in borosilicate glass

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HIGHLIGHTS

- The vitrification process was employed for immobilization of spent ion exchange (IEX) resins.
- The volume reduction ratio (VRR) of the prepared glass wasteforms was investigated.
- The chemical stability of the prepared glass wasteforms were investigated using the PCT method.

ABSTRACT

The present study examined the vitrification of spent ion exchange (IEX) resins in a borosilicate glass matrix on the laboratory scale. For this purpose, the simulated spent IEX resin waste prepared by doping non-radioactive cobalt and cesium elements on a combination of cationic and anionic resins was employed. For glass wasteform preparation, heat-treated (at 150 °C) spent IEX resin was mixed with borosilicate glass ferrite in the range of 20-40 weight percent and then melted at 1200 °C. To evaluate the prepared glass wasteforms, their volume reduction ratio (VRR) and chemical stability were investigated. According to the obtained results, with the increase in the spent IEX resin loading, the volume reduction ratio of the final wasteform increases. However, in the samples with spent IEX resins loading higher than 30 wt.%, phase separation (white color containing insoluble sulfate) was observed on the surface of the glass. Investigations showed that glass wasteform containing 30 wt.% spent IEX resins provides the best conditions for waste immobilization. The relative volume reduction ratio of this sample was measured as 86.61%. The normalized leaching rate of cesium and cobalt from this wasteform was calculated as 7.43×10^{-5} and 6.93×10^{-5} $\mathrm{g.m^{-2}.day^{-1}}$, respectively, using the PCT method.

KEYWORDS
Ion Exchange Resin
Borosilicate Glass
Vitrification
Wasteform
Cesium
Cobalt

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1 Introduction

With the rapid development of the nuclear industry in IRAN, a significant amount of radioactive waste has been generated. In terms of volume, the spent ion exchange (IEX) resins constitute a major portion of solid radioactive waste produced in the nuclear industry. IEX resins are employed to purify large volumes of hazardous radioactive effluent in the nuclear industry. However, their only limitation is the capacity of the resin to absorb different radionuclides. In such cases, the spent IEX resins need to be regenerated or replaced with fresh resins. However, spent IEX resins are not usually regenerated but are stored for disposal after use, as IEX resin regeneration is not cost-effective compared to their replacement. Even though the organic IEX resins used for this process are highly effective in removing radionuclides from wastewater, they are themselves problematic to immobilize or safely store. As a result, a large and growing inventory of this type of waste now exists globally, which requires a route for disposal.

Spent IEX resins produced in the Tehran research reactor are known as one of the most important radioactive waste streams that need to be managed in the country. These spent IEX resins are produced during the purification process of Tehran research reactor wastewater streams containing water-soluble radionuclides such as strontium-90, cobalt-60, cesium-137, etc. These spent IEX resins are classified as low- and intermediate-level radioactive waste.

Various methods for the management of spent IEX resins have been investigated (Cicero-Herman, 2002; Sheng, 2004). However, among the investigated meth-

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Table 1: The results of doping cesium on the clean resin.

Radionuclide	Loading wt.%	Initial Concentration (ppm)	Final Concentration (ppm)	Absorption Capacity (mg.g^{-1} IEX Resin)
Cesium-137	4.13	6900	4620	43
Cobalt-60	1.57	1258	409	16

ods, the immobilization of these wastes in a glass matrix in a way that incorporates the radionuclides into the macro- and micro-structure of the final wasteform has several advantages over other immobilization methods (Abbasi et al., 2020, 2024; Fayezi et al., 2023). The foremost of these advantages is that the vitrification method reduces the volume of the spent IEX resins by a significant factor through organic destruction, moisture evaporation, and reduction in porosity (Jantzen et al., 1995). This sets it apart from other methods available that increase the volume of material requiring disposal, such as cementation. Alongside this benefit, the high chemical resistance of glass allows the final wasteform to remain stable in corrosive environments for thousands or even millions of years, and it is also less susceptible to damage from radiation, chemical damage, and mechanical damage than other available wasteforms (Ojovan et al., 2019). Consequently, the disposal of vitrified wasteform can be achieved without additional engineering barriers in near-surface disposal and diverse geological environments, enabling the transportation and long-term storage of glassy wasteform due to its strength and long-term safety in waste repositories. In the United States, South Korea, Switzerland, Russia, England, and Ukraine, vitrification has been considered an alternative method to cementation for more than 40 years, even for low-level waste (LLW) (Ojovan and Lee, 2011). Generally, borosilicate glasses are the primary choice globally for immobilizing both high-level and intermediate- to low-level radioactive waste.

Hamodi and Iqbal (Hamodi and Iqbal, 2009) carried out research to determine the appropriate glass formulation for the immobilization of spent ion exchange resins from a PWR reactor, with an emphasis on the leaching of cobalt-60 and cesium-137 radionuclides. Comprehensive experimental and analytical data were presented in this work, demonstrating that the vitrification can reduce the volume of spent ion exchange resins while maintaining the required wasteform safety levels. (Rohyiza et al., 2013) employed the thermal treatment and ash vitrification methods to successfully immobilize the spent ion exchange resin from the PUSPATI TRIGA reactor of Nuclear Malaysia. They combusted spent ion exchange resins in a lab-scale combustor and vitrified the resulting ash with the addition of glass cullet powder as matrix material in a high temperature furnace.

McGann (McGann, 2014) in the Department of Materials Science and Engineering at the University of Sheffield worked on the development of glass compositions for the vitrification of spent ion exchange resin waste from the nuclear industry. According to their findings, ZnO-containing alkali alkaline-earth silicate glass compositions are suitable candidates for immobilization of spent ion exchange resins, with the ability to achieve high Cs retention. The present study was performed to investigate the immobilization of Tehran Research Reactor's spent IEX resin in a borosilicate glass matrix on a laboratory scale.

2 Experimental

2.1 *Materials and equipments*

All chemicals employed in this study were prepared and utilized in laboratory-grade purity. The IEX resin was provided by the Tehran Research Reactor. The cationic resin was Purolite C-100, which is a sulfonic acid-based resin in sodium form and the anionic resin was Purolite A-400, which is an ammonium-based resin in chloride form. A magnetic stirrer model MSH-20A from Switzerland was used for the IEX resin impregnation process. A laboratory scale with a measurement accuracy of one ten-thousandth, model AS 220 R2 PLUS from RADWAG Company was employed for weighing the samples. Also, a density kit, model KIT 85, was used for density measurements. The cesium concentration in the leachate samples was determined using an atomic absorption spectrometer (AAS) model SPECTRA AA200 from Germany. Scanning electron microscope (SEM) model EVO 18 made in Germany was employed for imaging of the prepared wasteforms and EDX and EBSD analysis. A furnace model F11L-1250 made by AzarFurnace company (Iran) was used to melt the samples. An oven model ACE400L made by ATRA company (Iran) was used for the leaching test.

2.2 *Spent IEX resin preparation*

Initially, the IEX resin impregnation process was carried out with non-radioactive cesium and cobalt. For this purpose, a solution containing specific amounts of cesium nitrate ($CsNo_3$) and cobalt nitrate ($Co(No_3)_2$) was prepared. The solution was then placed in contact with 300 grams of ion exchane resin (150 grams of cationic resin and 150 grams of anionic resin) for 48 hours at room temperature and stirred at a speed of 180 rpm using the MSH-20A magnetic stirrer. This allowed the cesium and cobalt to be loaded onto the ion exchane resin completely. The results of the cesium and cobalt doping process on the IEX resin are shown in Table 1. In the next step, the loaded IEX resin was placed in a furnace and subjected to a temperature of 150 °C for thermal treatment. This thermal treatment caused the IEX resin to lose 50% of its weight, primarily in the form of moisture. Additionally, the thermal treatment allowed the IEX resin to become compatible and homogenized with the borosilicate glass composition during the melting process.

Table 2: The composition of borosilicate glass ferrite.

SiO_2	B_2O_3	Al_2O_3	Fe_2O_3	BaO	CaO	Na_2O	Li_2O	ZnO
51.72	0.38	2.23	2.92	23.22	2.08	5.64	7.81	5.00

Table 3: Weight percentage of borosilicate glass frrite and simulated waste resin.

Temperature (°C)	1200	1200	1200	1200	1200
Glass frrite (wt.%)	80% (16 g)	75% (15 g)	70% (14 g)	65% (13 g)	60% (12 g)
Resin (wt.%)	20% (4 g)	25% (5 g)	30% (6 g)	35% (7 g)	40% (8 g)

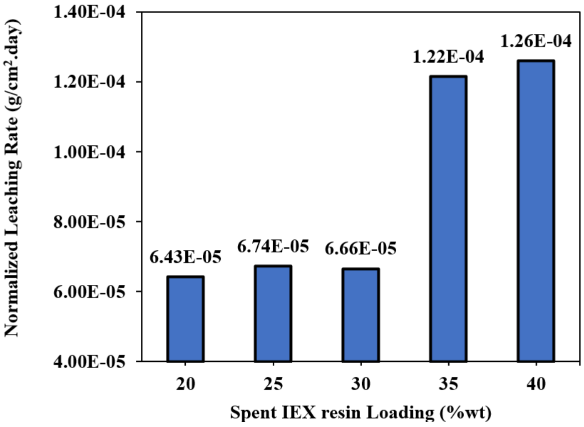

Figure 1: Cobalt normalized leaching rate as a function spent IEX resin loading at 1200 °C.

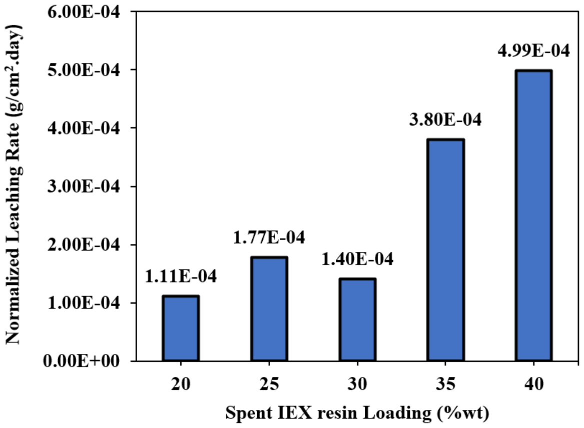

Figure 2: Cesium normalized leaching rate as a function of spent IEX resin loading at 1200 °C.

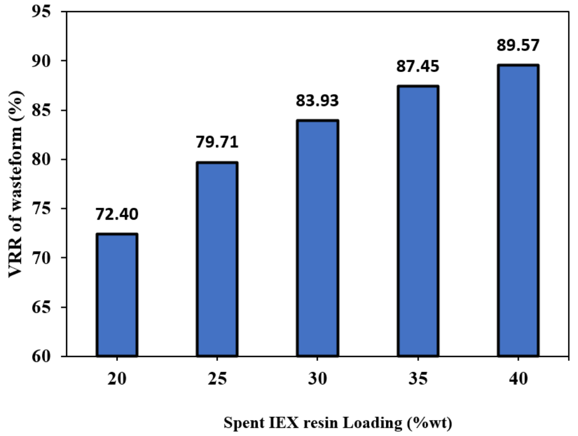

Figure 3: The VRR as a function of spent IEX resin loading.

2.3 Spent IEX resin immobilization process

To create the glassy wasteform, a mixture consisting of 20 grams of borosilicate glass and the waste resin was considered. Table 2 shows the composition of the borosilicate glass used in this study. The characteristics of the prepared wasteforms are reported in Table 3. Subsequently, the glass frrite-simulated resin waste mixture was poured into an alumina crucible and placed inside the furnace. The rate of temperature increase was also 5 degrees per minute. The glass mixture eventually reached a temperature of 1200 °C and was held at this temperature for 3 hours. After the melting process was complete, the alumina crucible was removed from the furnace and placed in a preheated annealing furnace at a temperature of 500 °C to allow the annealing process of the wasteform to proceed slowly.

2.4 Glass wasteform characterization

In order to determine the volume reduction ratio in the spent IEX resin vitrification process, the density of the vitrified wasteform was first measured using the Archimedes method and using a density measurement device connected to an analytical scale. The initial volume of the wasteform was also measured using a graduated cylinder. By knowing the volume of the wasteform before and after immobilization, the volume reduction ratio (VRR) was calculated according to Eq. (1):

$$VRR = \frac{V_i - V_f}{V_i} \times 100 \tag{1}$$

For determining the normalized leaching rate of cesium and cobalt from final wasteforms, leaching tests were performed by PCT (Product Consistency Test) standard test method (ASTM, 2002). For this purpose, the produced glass wasteforms were powdered in the range of 75-150 μm and placed in contact with the leaching solution of distilled water in a closed PTFE container and placed in the oven for 7 days at 90 °C. Then, the samples were filtered by filter paper and the cesium and cobalt concentration in the solution was measured. The normalized leaching rate (NLR) was calculated using Eq. (2):

$$NLR = \frac{C}{(f)(SA/V)(t)} \tag{2}$$

In this equation, C is the concentration of cesium and cobalt in the leachate solution ($g.L^{-1}$), f is the weight

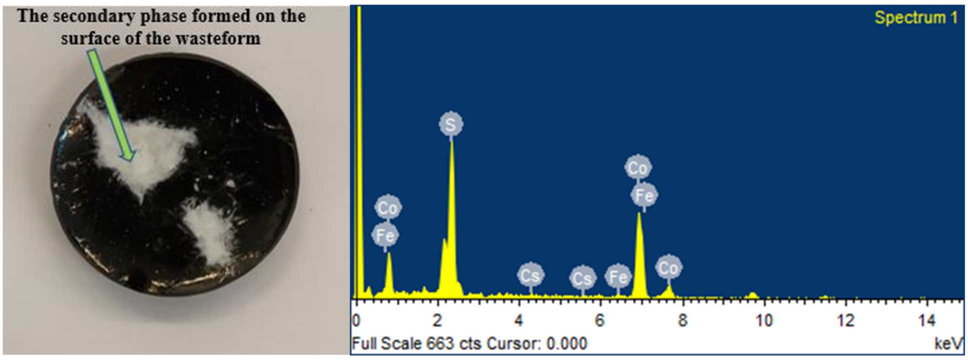

Figure 4: The image and EDX analysis of phase separation zone in the wasteform containing 35 wt.% spent IEX resin.

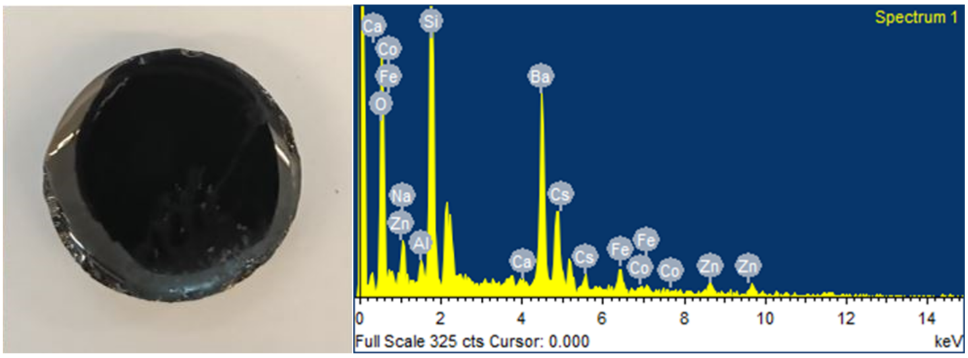

Figure 5: The image and EDX analysis of the wasteform containing 30 wt.% spent IEX resin.

fraction of cesium and cobalt in the initial matrix, SA/V is the surface area ratio of the glass samples to the volume of the leachate solution in terms of $cm^2.L^{-1}$, and t is the leaching time in days.

3 Results and Discussion

Normalized leaching rate of cesium and cobalt as a function of spent IEX resin waste loading at 1200 °C are shown in Figs. 1 and 2, respectively.

As observed from Figs. 1 and 2, with an increase in the waste loading, the amount of leached cesium and cobalt from the wasteforms are increase, therefor the immobilization of cesium and cobalt in the wasteform matrix decrease. Additionally, the values corresponding to the volume reduction ratio of different wasteforms are reported in Fig. 3, indicating the effect of the waste loading on the volume reduction ratio at a temperature of 1200 °C.

The results indicates that with the increase in the spent IEX resin loading, the volume reduction ratio of the final wasteform increases.

The appearance of the wasteforms showed that phase separation occurs on the surface of samples with waste loading of 35 wt%, as shown in Fig. 4. Moreover, in order to characterize the produced wasteforms and investigate the micro-structure of the wasteforms and the secondary phase generated, scanning electron microscopy (SEM) images of the samples were obtained and showed that this phase contains high amounts of sulfur, iron, and cesium.

Investigations revealed that glass wasteform containing 30 wt.% spent IEX resins provides the best conditions for waste immobilization. Figure 5 presents the image and EDX analysis of the wasteform containing 30 wt.% spent IEX resin

4 Conclusions

The results of this study show that the borosilicate glass matrix is suitable for immobilizing the spent IEX resin of the Tehran Research Reactor up to 30 wt.% at a temperature of 1200 °C. Under these conditions the relative volume reduction ratio of this final wasteform sample was measured as 86.61%. The normalized leaching rate of cesium and cobalt from this wasteform was calculated as 7.43×10^{-5} and 6.93×10^{-5} $g.m^{-2}$.day, respectively, which is considered an acceptable value based on the available literature. Additionally, at higher waste loadings, a secondary phase containing significant amounts of sulfur, cesium, and iron was formed on the surface of the borosilicate glass wasteform. The results of the glass waste leaching test revealed that this phase has a high dissolution capability when in contact with water, resulting in a significant increase in cesium and cobalt leaching results.

Conflict of Interest

The authors declare no potential conflict of interest regarding the publication of this work.

References

Abbasi, A., Avanes, A., Davarkhah, R., et al. (2024). Efficient sorption and secure immobilization of strontium ions onto nanoporous alumino-borosilicate as a new matrix. *Scientific Reports*, 14(1):21617.

Abbasi, A., Davarkhah, R., Avanes, A., et al. (2020). Development of nanoporous alumino-borosilicate as a novel matrix for the sorption and stable immobilization of cesium ions. *Journal of Inorganic and Organometallic Polymers and Materials*, 30:369–378.

ASTM (2002). Standard test methods for determining chemical durability of nuclear, hazardous, and mixed waste glasses and multiphase glass ceramics: the product consistency test (PCT). *ASTM International, West Conshohocken*.

Cicero-Herman, C. A. (2002). Commercial ion exchange resin vitrification studies. Technical report, Savannah River Site (SRS), Aiken, SC (United States).

Fayezi, M., Shiri-Yekta, Z., Sepehrian, H., et al. (2023). Adsorption and safe immobilization of sr ions in modified zeolite matrices. *Scientific Reports*, 13(1):19087.

Hamodi, N. H. and Iqbal, Y. (2009). Immobilization of spent ion exchange resin arising from nuclear power plants: an introduction. *Journal of Pakistani Material Society*, 3(1):7–18.

Jantzen, C. M., Peeler, D. K., and Cicero, C. (1995). Vitrification of ion-exchange (IEX) resins: Advantages and technical challenges. Technical report, Savannah River Site (SRS), Aiken, SC (United States).

McGann, O. J. (2014). *The Development of Glass Compositions for the Vitrification of Ion Exchange Resin Wastes from the Nuclear Industry*. PhD thesis, University of Sheffield.

Ojovan, M. I. and Lee, W. E. (2011). Glassy wasteforms for nuclear waste immobilization. *Metallurgical and Materials Transactions A*, 42:837–851.

Ojovan, M. I., Lee, W. E., and Kalmykov, S. N. (2019). *An introduction to nuclear waste immobilisation*. Elsevier.

Rohyiza, B., Zalina, L., Mohd, A. W. Y., et al. (2013). Feasibility study on vitrification of spent ion exchange resins from TRIGA Reactor Malaysia. *Journal of Nuclear and Related Technologies*, 10(2):30–42.

Sheng, J. (2004). Vitrification of a spent ion exchanged resin from a nuclear power plant. *Glass Technology*, 45(3):153–156.

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