# CONDITIONING AND IMMOBILIZATION OF

# INACTIVE ION EXCHANGE RESINS IN

# THERMOPLASTIC POLYMERS

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**Abstract**

Spent ion exchange resins (IER), must be conditioned for storage and disposal, in order to reduce the possibility of dispersion, elution and diffusion of radioactive species to the environment. The immobilization of 0, 10, 20 and 30 wt% of dry cation exchange resins (CER), in linear low-density polyethylene (LLDPE), high impact polystyrene (HIPS) and recycled polyethylene (RPE), was studied. Each waste form was studied as non-irradiated and 100 kGy 60Co gamma irradiated. The properties studied were swelling, mechanical properties and leaching resistance. The leaching tests were carried out with CER loaded with a non-radioactive solution of Cs+, Co2+, Ni2+ and Sr2+ measured by Total Reflection X-ray Fluorescence (TXRF). For CER immobilized in LLDPE, an increase in flexural modulus was observed when CER contents increased as well as irradiated compared to non-irradiated samples. With RPE, no differences were observed in flexural modulus when the CER contents increased or for the irradiated samples. HIPS showed a decrease in the flexural modulus when the CER contents increased. However, no difference was observed between the irradiated and non-irradiated samples. The cumulative leached fraction values were between 0.008 and 0.006 for Cs+ and 0.004 and 0.001 for Co2+, Ni2+ and Sr2+, respectively, for CER in LLDPE and RPE. An increase in the leaching of the non-irradiated specimens compared to the irradiated ones was observed for LLDPE. This effect was not observed for RPE. The cumulative fraction leached for HIPS was 0.015 for Cs+ and 0.002 for the other cations, being higher in the irradiated than in the non-irradiated samples. No swelling effect was observed for any condition and studied material. RPE showed the best performance as immobilizing matrix for CER in these conditions. Using recycled plastic in nuclear industry is an opportunity to include plastic waste as a resource in the context of a circular economy.

## INTRODUCTION

Polymeric ion exchange resins (IER), based on sulfonate- and quaternary amine-functionalized polystyrene divinyl benzene (PSdVB) copolymers, are used in nuclear power plants for the purification of water in the cooling system circuits [1-5]. Spent IER as radioactive waste represent a large volume of radioactive waste generated during the operation of these plants.

The resins accumulate fission products, neutron activation products and actinides such as uranium and transuranium elements from the nuclear fuel.

The management of spent IER as radioactive waste involves many activities like radiochemical characterization, storage, pre-treatment selection, treatment and conditioning processes, evaluation of the properties of the waste form and subsequent storage and/or disposal [1,6].

Many of the radioisotopes present in IER have long half-lives (t1/2), therefore it is necessary to isolate them for a long time to protect people and the environment from the adverse effects of the ionizing radiation. The long times periods involved in the storage and disposal make it necessary to treat and condition the waste in order to reduce the possibility of dispersion, elution and diffusion of radioactive species [1].

The immobilization matrixes that have been mainly used and continue to be developed, for low and intermediate level waste such as IER, are cement, bitumen, epoxy resins and thermoplastic polymers [7]. A suitable immobilizing matrix must be chosen taking into account their availability, low cost, volumetric efficiency, simplicity of use and the properties of the waste form [8,9]. Considering these characteristics, a waste form must present low leaching rate, chemical and thermal stability, mechanical and radiation resistance, low swelling and solubility and it must also be non-biodegradable [1].

Cement is the most studied and used matrix for the immobilization of this kind of radioactive waste. This material has excellent mechanical properties, physical and thermal stability, as well as being inexpensive and widely available. One of the best-known disadvantages of cement is its high porosity, which would also allow much greater leaching than other immobilizing matrixes. However, many formulations and combinations with other materials have been studied to improve these characteristics [10]. One main disadvantage associated with cement to immobilize IER is its low capacity for incorporation of high content IER. As much as 20 wt% of resin immobilizations have been achieved but with a 6-fold increase in the waste form volume [11,12]. Although most immobilization processes imply an increase in the volume of the conditioned waste, the low capacity of the cement and its weight make it necessary for other alternatives to be sought due to the costs associated with transportation.

Bitumen is another material that has been used as an immobilizing matrix. It is chemically resistant and allows greater resin immobilization and lower volume increase than cement. Although bitumen has less radiation stability, the waste form presents less leaching than cement. It has good rheological properties and plasticity, which allows deformation with less brittleness and rupture of the material [1,13,14].

Epoxy resin as thermosetting polymer has been widely studied for IER immobilization. Epoxy resins allow the immobilization up to 50 wt% IER, radiation resistance, leaching resistance, excellent mechanical properties and resistance to environmental degradation. Although, with excellent mechanical properties, the waste form could be brittle allowing the fracture of material. The high cost of epoxy resin limits their commercial application [2,5].

Thermoplastic polymers are other material used for the immobilization of IER. Compared with cementation, this process needs IER dehydration and the operation process and facilities are complex [10]. Leaching is less predominant in these polymers than in cement and they have a greater capacity to immobilize the resin than cement. These polymers are resistant to radiation but not as resistance as cement. Compared to bitumen, they have superior thermal and mechanical stability [1,15].

Polyethylene is one of the most widely used thermoplastics for immobilization of IER [16]. Depending on whether it is a high-density (HDPE) or low-density (LDPE) polyethylene, mechanical properties, swelling, flammability index, dissolution in solvents and other properties may change. LDPEs are preferred over HDPEs since they have a lower melting point, therefore they can be processed at lower temperatures, preventing the volatilization of certain radionuclides [8]. Some authors studied the performance of radioactive IERs immobilized in polyethylene. Their results showed that with 50 wt% of immobilized resin and with irradiation doses of 1000 kGy, no change in mechanical strength was observed [10,17]. The waste form showed no deformation by swelling, unlike bitumen products. Leaching was significantly less than other types of matrixes such as cement and bitumen [16,18].

Polystyrene is another thermoplastic available for immobilization processes. Researchers studied the immobilization of IER in this matrix. The waste form has shown good thermal resistance and leaching resistance, but high price and strict operation conditions [10].

Because of the brittleness of polystyrene, high impact polystyrene (HIPS) was studied. HIPS is a copolymer of polystyrene and polybutadiene rubber. The composition and morphology of this thermoplastic provides a good balance between stiffness and elasticity [19].

Due to efforts being made worldwide to reduce the environmental impact of plastics, the possibility of using recycled plastics (after being used or scrap) for the immobilization of this type of waste has been considered [20, 21]. However, the use of recycled plastics for immobilization of IER has not been enough developed so far. Research is needed in order to compare the behavior of recycled polymers with raw virgin materials, diversifying the existing options. In this way, the results will contribute to generate knowledge and technology which can be evaluated as a possible strategy for the treatment and conditioning of this kind of radioactive waste.

The use of recycled plastics in the treatment of radioactive waste could also be a strategy to reduce their environmental impact and a resource which can contribute to the circular economy of plastics.

In this work, the immobilization of cation exchange resins (CER) in virgin and recycled thermoplastic polymers was evaluated. Linear low-density polyethylene (LLDPE), recycled polyethylene (RPE) and high impact polystyrene (HIPS) were studied. The CERs were dried at 160 ° C to reduce the volume and to release the contained water to avoid the formation of bubbles during the immobilization process of thermoplastics. The different immobilizing polymeric matrixes/CER compositions were obtained by discontinuous process. Testing specimens were manufactured by thermo compression. Flexural mechanical properties, swelling and leaching capacity were evaluated in non-irradiated and 100 kGy 60Co gamma irradiated samples.

## materials and methods

Immobilization of Cation Exchange Resin (CER) in thermoplastics polymers was studied. The matrixes used were Linear Low-Density Polyethylene (LLDPE), High Impact Polystyrene (HIPS) and Recycled Polyethylene (RPE). The used CER were DOW AMBERLITE IR 120 Na with molecular structure of polystyrene crosslinked with divinylbenzene (PS-DVB) and sulfonic acid as functional groups. The resin total exchange capacity was 2.0 eq/L, shipping weight of 840 g/L and 28 % of humidity was determined by thermogravimetric analysis (TGA). CER were dried at 160 °C, not only to reduce the volume of waste but also as a necessary condition to immobilize in thermoplastics polymers, in order to avoid the production of bubble during the process and the manufacture of defective specimens [22].

Waste forms of 0, 10, 20 y 30 wt% immobilized CER in LLDPE, HIPS and RPE were manufactured. CER loaded with Cs+, Co2+, Ni2+ and Sr2+ cations from simulating solution were used and immobilized at 30 wt% to carry out the leaching tests. The different waste forms were irradiated at 100 kGy in 60Co gamma source to simulate the radiation effect of the radioactive waste.

Leaching, flexural mechanical tests and swelling were analyzed from different waste form, non-irradiated and irradiated.

### Load of CER with cations simulating solution

CER were loaded with simulating solutions of stable isotopes of Cs+, Co2+, Ni2+ and Sr2+ cations. To carry out this assay, 40 g of CER were mixed with 1L of simulating solution 15 mM in each of cation in 0,1 M HNO3, during 30 minutes and 70 rpm to get a suitable load.

The cations content was measured at the beginning and after 30 min contact with CER by Total Reflection X-Ray Fluorescence (TXRF) to define the loaded percent.

### Waste forms and testing specimens preparation

Blends of thermoplastics polymers and CER were made in a Brabender Plasticorder discontinuous mixer machine. Waste forms of LLDPE, HIPS and RPE with 0, 10, 20 and 30 wt% of CER, dry and unloaded, were made to the flexural assays. Waste forms with 30 wt% of dry and cation loaded CER were made to evaluate leaching and swelling material. Processing temperatures were 180 °C for LLDPE and RPE, and 220 °C for HIPS were used.

From each waste forms, rectangular specimens were obtained by thermos molding and thermocompression, in hydraulic press. The specimen’s measures were 100 mm x 10 mm x 3 mm.

### Samples Irradiation

The specimens prepared as described before were subjected to gamma radiation dose of 100 kGy in 60Co gamma ray source, during 4 h, at 15 cm from source and rate dose of 23.8 kGy/h. Non-irradiated and irradiated samples were used for comparison purposes.

### Flexural mechanical testing

Ten rectangular specimens (100 mm x 10 mm x 3 mm) of each CER/polymer composition were analyzed following the recommendation of ASTM D790 – 03 (2003) standard: *“Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials”* [23].

These tests were carried out in mechanical testing machine (Instron-5985) with a load cell of 1 kN. The specimen rests on two supports at 60 mm of distance from each other and the third point is loaded on the midway between the supports. The rate of crosshead motion was 2 mm/min until 10 mm of maximum strain.

### Leaching tests

Leaching tests were carried out under recommendation of ANSI/ ANS-16.1-1986 standard: *“American National Standard Measurement of the Leachability of solidified Low-Level Radioactive Waste by a Short-Term Test Procedure”* [24].

Specimens of LLDPE, RPE and HIPS with CER 30 wt%, dry and loaded with Cs+, Co2+, Ni2+ and Sr2+ cations obtained as was described before, non-irradiated and irradiated, were analyzed. Surface area of these samples were 4 cm2 (1,2 cm x 1,2 cm x 0,3 cm). The test was made by triplicate. The samples were mixed with 40 ml of deionized water used as leaching solution, at room temperature and 70 rpm. Leachate was removed and renew at different times (30s, 7, 24, 48, 72, 96 and 120 h). Cations from each leachate were measured by TXRF and the results are expressed as cumulative leached fraction through 120 h of experiment.

### Swelling tests

Swelling was analyzed at the end of the leaching test. Specimens of LLDPE, RPE and HIPS with CER 30 wt%, dry and loaded with Cs+, Co2+, Ni2+ and Sr2+ cations, non-irradiated and irradiated, were analyzed. These samples were immersed in deionized water during 120 h. Weight and volume were measured at the beginning (t0) and at the end of assay (t120). The test was run by triplicate for each condition.

## RESULTS AND DISCUSSION

Loading process with Cs+, Co2+, Ni2+ and Sr2+ reached around 63% of the full CER capacity. The volume of the dried CER at 160 °C was reduced by about 50 vol% and a reduction of 28% of weight because of loss of water.

The stress (MPa) vs strain (mm) curves for flexural mechanical test, for each specimen with different combination CER/ polymer, non-irradiated and irradiated, were evaluated (Fig.1).

LLDPE waste forms irradiated curves (Fig. 1 - right) are above the non-irradiated ones (Fig. 1 - left) for all immobilized CER compositions. This could be due to cross-linking of the LLDPE matrix due to the effect of radiation. In addition, for both irradiated and non-irradiated samples, the stress increases as the amounts of CER increase. On the other hand, RPE did not show significant differences between the irradiated and non-irradiated samples and neither between the different amounts of immobilized CER. LLDPE and RPE specimens were not broken during the test (Fig.1).

HIPS waste forms showed no differences between the irradiated and non-irradiated samples for each amount of immobilized CER. All HIPS specimens tested broke before the end of the test at 10 mm of strain. The stress and strain values at break point were lower as the CER content increased, for both the non-irradiated and irradiated samples. The strain value at break point was 6 mm for HIPS without resin, strain between 5-6 mm for HIPS with CER 10 wt%, 4-5 mm for CER 20 wt% and between 3-4 mm for CER 30 wt% (Fig.1).

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*Fig. 1: Stress (MPa) vs Strain (mm) graph for the waste forms of LLDPE, RPE and HIPS with CER 0, 10, 20 and 30 wt%, non-irradiated (left) and**60Co gamma irradiated (right).*

Maximum load and flexural modulus as a function of CER content for the studied matrixes is showed in Fig. 2. For LLDPE / CER, an increase in the values of maximum load (at 10 mm of strain) and flexural modulus was observed with increasing CER content, both for the irradiated and non-irradiated samples. The irradiated samples presented a higher maximum load and modulus compared to the non-irradiated ones.

For the materials obtained from RPE, the maximum load and flexural modulus values did not show significant differences as the CER content increased, both for the irradiated and non-irradiated samples. Different behavior was obtained for HIPS/ CER mixtures, non- irradiated and irradiated, where a decrease in both maximum load (less than 10 mm of strain) and flexural modulus was observed, as the CER content increased.



*Fig. 2:* *Maximum load (N) vs Immobilized resin (%) (left). Flexural Modulus (MPa) vs Immobilized resin (%) (right), for different non-irradiated and 60Co gamma irradiated samples.*

Fig. 3 showed the results of the leaching test of Cs+, Co2+, Ni2+ and Sr2+ for each polymer/CER 30 wt%, non-irradiated (0 kGy) and 60Co gamma irradiated (100 kGy).

For LLDPE/CER 30 wt% samples, cumulative leached fraction of Cs+ reached values less than 0.008 after 120 h for both the non-irradiated and the irradiated samples. Although a steady state was not reached in this test time, the leaching rate decreases significantly after 24 h. These results are promising for this type of high mobility cation [4, 25]. Cumulative leached fraction of Co2+, Ni2+ and Sr2+ reached values of 0.0035 and 0.0025 for the non-irradiated and irradiated samples, respectively. Steady state for these cations was reached at 24 h for non-irradiated and 7 h for the irradiated samples. The cumulative leaching fraction curves showed a trend to greater leaching for the non-irradiated than for the irradiated ones, for each cation (Fig. 3). This result could be a consequence of the crosslinking produced by gamma radiation on the polymer structure. This effect was also observed in the results of the flexural mechanical tests.

For RPE/CER 30 wt% samples, cumulative leached fraction of Cs+ reached values less than 0.006 and values as low as 0.001 for Co2+, Ni2+ and Sr2+, for non-irradiated and irradiated samples, after 120 h of testing. The steady state for Cs+ began in the range of 7 and 24 h, however, for the other cations at 7 h it was already reached. Unlike LLDPE, the curves of the non-irradiated and irradiated samples were overlapped for Co2+, Ni2+ and Sr2+. The cumulative leached fraction of Cs+ curves for non-irradiated presented slightly higher values than the curves of the irradiated samples (Fig. 3). As observed in the results of mechanical properties, RPE is a material that structurally allowed to adapt to the potential effects caused by radiation.

HIPS presented a different cation leaching behavior compared to the polyethylene matrices studied. In this case, it was observed that the cumulative leached fraction curves of the irradiated samples showed higher values than the curves of the non-irradiated samples, suggesting a trend to greater leaching. The Cs+ reached a cumulative leached fraction of 0.008 and 0.014 at 120 h of testing in the non-irradiated and irradiated samples, respectively. The Co2+, Ni2+ and Sr2+ presented values between 0.001 and 0.002 for the non-irradiated and the irradiated samples, reaching the steady-state after 7 h of the test (Fig. 3). The greater leaching observed after irradiated HIPS samples compared to non-irradiated conditions could be related to the formation of cracks in the polybutadiene phase, promoting the migration of cations from the polymeric matrix to the leaching solution [19].

The difference in Cs+ leaching among the different non-irradiated materials was small but Cs+ cumulative leached fraction of HIPS was higher than the others material for irradiated samples. LLDPE was the material with the highest cumulative leached fraction for Co2+, Ni2+ and Sr2+, both in non-irradiated and irradiated samples. Cumulative leached fraction of Co2+, Ni2+ and Sr2+ for non-irradiated RPE and HIPS were very close but the leaching in LLDPE was almost doubled (Fig. 3).

For Co2+, Ni2+ and Sr2+ a steady-state was achieved before of 120 h of leaching for all non-irradiated and irradiated materials. For Cs+ the results were different, probably due to highly mobility of this cation [4]. However, as was mentioned before the curves show a trend where steady-state will be reached in a short time, not exceeding 0.02 of the cumulative leached fraction.

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*Fig. 3: Leaching of Cs+, Co2+, Ni2+ and Sr2+ for each polymer/CER 30 wt%, non-irradiated (0 kGy) and 60Co gamma irradiated (100 kGy).*

The differences observed in cumulative leached fraction for the studied materials could be attributed to the different composition and morphologies of the polymeric networks. As well as a result of the rearrangement of the polymeric chains after the immobilization process and due to the impact of the gamma radiation on the composite material (polymeric matrix + CER).

RPE as a recycled material, has been subjected to at least two temperature processes. This could have produced rearrangements in the polymeric network, modifying the leaching capability. This may explain why no significant difference was observed between non-irradiated and irradiated samples. LLDPE as virgin raw material, has not been subjected to a re-processing operation, so the differences between non-irradiated and irradiated samples were more significant. HIPS is a copolymer of polystyrene and polybutadiene rubber. Gamma radiation produced a particular effect on each molecular structure. Butadiene is more sensitive to cracking than polystyrene and this could be the reason for higher cumulative leaching values in irradiated than non-irradiated HIPS/CER 30 wt%, as described by some authors [19].

The swelling of composite materials (polymer-CER) depends on the hydrophilic characteristics of the polymer and the content of CER in the waste forms. Water swelling of an ion exchange resin is due primarily to the hydration of the fixed ionic groups [26].

As mentioned before, swelling was analyzed at the end of the leaching assay. Weight and volume were determined at t=0 and t=120 h.

No significant differences were observed in the volume of the LLDPE and RPE specimens with CER 30 wt%, for non-irradiated and irradiated samples after 120 h submerge in water. The swelling value obtained was less than 1%. According to several authors, this result implies that there is no swelling in the polymer matrix studied, under the conditions of this test [27]. In the case of the weight change measurement, an increase of 2% was observed for the non-irradiated and irradiated samples. These values are below those mentioned by some authors where they consider 8% swelling as moderate values [28]. No volume changes were observed for non-irradiated and irradiated HIPS specimens. However, 1,5% weight increase was observed for irradiated samples. As previously mentioned, this increase is considered not significant for matrix swelling characterization. Many authors [19, 29] have described that low irradiation doses produce crosslinks in polybutadiene but at doses above 100 kGy from a 60Co gamma source can cause cracks in the material. These microcracks could contribute to the increase in weight due to water absorption of the irradiated samples over the non-irradiated in HIPS.

These volume and weight changes showed that the stability or long-term durability of the CER immobilized in thermoplastic polymers studied should not be affected.

## Conclusions

The results obtained in the present work showed that RPE is seen as the most suitable matrix for the immobilization of the CER loaded with Cs+, Co2+, Ni2+ and Sr2+ cations. RPE/ CER 30 wt% waste forms showed no significant difference in flexural mechanical properties, leaching and swelling between non-irradiated and 60Co gamma irradiated samples. Furthermore, these properties were not affected by different amount of CER immobilized in RPE.

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