

## Results from Waste ERASER: Testing the Complete Treatment Process from Chemical Decomposition to Final Conditioned Waste Package

Céline Boulet, Wolfgang Schuch, Klaus Büttner, Thomas Fishedick, Robert Lehr, Sven Nothvogel

Framatome GmbH

Paul-Gossen-Straße 100, 91052 Erlangen, Germany

[sven.nothvogel@framatome.com](mailto:sven.nothvogel@framatome.com), [celine.boulet@framatome.com](mailto:celine.boulet@framatome.com),  
[wolfgang.schuch@framatome.com](mailto:wolfgang.schuch@framatome.com), [klaus.buettner@framatome.com](mailto:klaus.buettner@framatome.com),  
[thomas.fishedick@framatome.com](mailto:thomas.fishedick@framatome.com), [robert.lehr@framatome.com](mailto:robert.lehr@framatome.com)

### ABSTRACT

Solidification and in particular cementation of spent ion exchange (IEX) resins often faces challenges, such as a low incorporation rate into the matrix, beads swelling, presence of combustible organic content and matrix inhomogeneity. The innovative process of spent IEX resin liquefaction avoids the common problems associated with IEX resin cementation. The liquefaction process reduces the organic content and results in a homogeneous distribution of radioactivity in the final waste form. The higher level of possible incorporation of treated IEX in the conditioned waste, e.g. cement matrix, offers significant cost-saving potential. In some cases, reducing the organic content of spent IEX resins is a requirement for disposal at repositories.

To address this issue, Framatome developed the Waste ERASER (Extended Volume Reduction by Advanced Treatment of Spent Ion Exchange Resins – formerly SPRINT) process for treating spent IEX resin including different possibilities of final conditioning. Initial on-site demonstration trials were conducted in 2020 at a nuclear power plant (NPP), involving the liquefaction of radioactive spent IEX resins from operation and cementation of the resulting liquefied resins. The produced specimens were then evaluated for leaching behavior and compressive strength according to the Swiss waste acceptance criteria.

Following successful demonstration trials, Framatome designed, manufactured, and commissioned a semi-industrial scale pilot plant at Framatome's site in Karlstein, Germany. This pilot plant incorporated experience feedback gained from the on-site NPP trials and demonstrates how a flexible and simple liquefaction plant could be implemented in a NPP using a modular design.

Framatome has initiated a research and development project, partially funded by the German Ministry of Education and Research, utilizing its semi-industrial scale pilot plant. This R&D project is divided into two main phases. The first phase successfully focused on gaining operational experience with the process, emphasizing parameters for safe and efficient operation. The second phase focused on the immobilization of the resulting liquid concentrates using different matrices and recipes.

After the completion of both phases in 2025, the results will be presented. Different IEX resins and several simulated ion loads on the resins have been tested. The simulated ion loads have been selected to represent IEX resins used for wastewater treatment during operation and also IEX resins which are used during a full system decontamination.

The liquefied IEX resins derived from the pilot plant were immobilized using different cement formulations, geopolymer formulations and drying techniques. Emphasis was placed on ensuring volume optimization and alignment with international waste acceptance criteria.

**Keywords:** spent resin, immobilization, cementation, geopolymerization, volume optimization

## 1 INTRODUCTION

During operation of almost all types of nuclear power plants, spent ion exchange resins (IEX) are generated and must be conditioned and disposed of as radioactive waste. The main used conditioning technique to produce a stable matrix that meets final and intermediate storage requirements is cementation, resulting in a factor of around four of volume increase (depending on the respective recipe) and therefore corresponding volume-specific costs for final disposal. In addition to high disposal costs, cementation of IEX is associated with waste form uniformity and durability problems due to IEX inherent characteristics.

Cementation of spent IEX is often accompanied by difficulties, such as the transport of the IEX, accurate dosing of the IEX during the mixing process, difficulties to draw a homogeneous sample and unstable matrices due to swelling effects, hot spots in the cement matrix, and ion exchange between the IEX and the hydrated cement matrix. This can lead to cracks and weakening of the cement matrix. To achieve a stable matrix, the resin loading must according to Framatome's experience be kept low.

Framatome started several years ago to develop a method to deal with the problematic IEX matrix (polystyrene-divinylbenzene matrix). Based on the Fenton process, a chemical process was developed that can destroy the matrix of the IEX and significantly reducing other organic components on the IEX. The so-called "liquefaction" of the IEX destroys the polymer matrix and allows homogenization of radionuclides in the resulting aqueous solution, thus avoiding the problems described above. This also enables a significantly higher incorporation rate in the final waste form.

Framatome's method is mild (at atmospheric pressure, temperature  $\ll 100^{\circ}\text{C}$ ) and robust. If necessary, the organic content can be reduced below 100 ppm by an additional oxidation process (advanced electrochemical oxidation). If C-14 forms a significant issue with the spent IEX, most of the C-14 bound on the IEX can be removed and captured before the liquefaction process by another process developed by Framatome.

First on-site demonstration trials were conducted in 2020 at NPP Gösgen, Switzerland including liquefaction of representative spent IEX, removal of process water by vacuum evaporation, and cementation of the resulting evaporator concentrate. Subsequently, leaching behavior and compressive strength were analyzed in accordance with Swiss waste acceptance criteria.

Based on these results, Framatome developed an intermediate scale pilot plant for chemical treatment of spent resin (so-called liquefaction of IEX) which was manufactured and set-up in 2022 at Framatome's site in Karlstein, Germany. The pilot plant was commissioned in 2023, followed by further non-radioactive testing to adjust control parameters and settings. In 2024, the second phase of the project focused on the solidification of the liquefied IEX via different conditioning methods: cementation, geopolymerization and drying with a rotating drum drier. Emphasis was placed on optimizing the conditioning process to ensure volume minimization and alignment with international waste acceptance criteria.

## 2 LIQUEFACTION PROCESS

Between 2023 and 2025, more than 30 resin liquefaction tests were carried out on ion exchanger material using the pilot plant. For this purpose, IEX were simulated using Lewatit S200 and M800 (exemplary for operational resins) as well as Purolite NRW100 and NRW400 (exemplary for resin used in decontamination processes) and loaded accordingly. The ion exchange

material was treated batchwise in charges of 15 – 30 litres (maximum capacity for resins of the pilot plant). The liquefaction process is a semi-batch-wise process with a continuous feed of hydrogen peroxide. The hydrogen peroxide feed is increased over the course of time as shown in Figure 1.

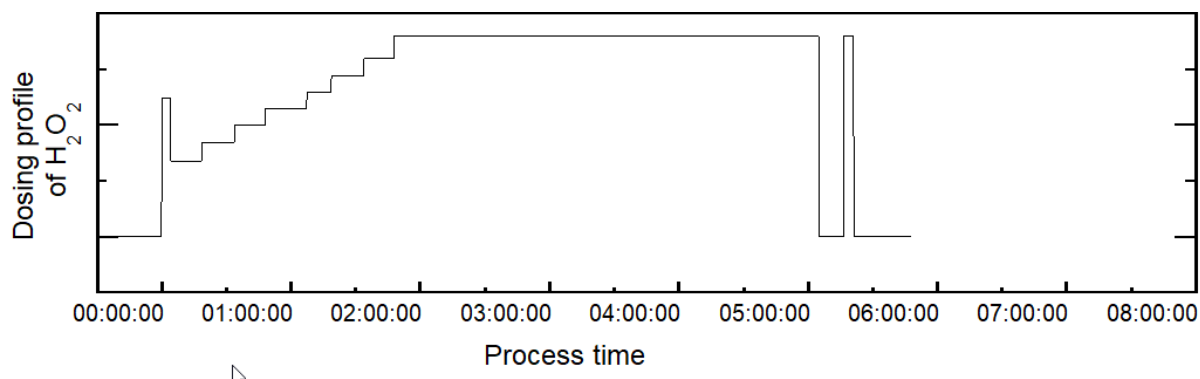


Figure 1: Dosing concept of H<sub>2</sub>O<sub>2</sub> for spent resin decomposition using a dosing ramp

The organic IEX matrix is destroyed by the process resulting in a liquid solution and as shown in Figure 2. The resulting liquid waste can be either dried directly (for example using a rotating drum drier) or concentrated via evaporation for a further conditioning step.



Figure 2: IEX destruction over time (left: initial IEX, right: destroyed IEX)

The volume after termination of the liquefaction process of 22.5 liters IEX was about 235 liters. A dry residue (at 120°C) of 3.5% was determined in the liquid solution after the test. The density of this solution was approximately 1.03 kg/L resulting in a dry residue of about 8.5 kg. Furthermore, the total organic carbon content was determined to be 1525 ppm resulting in a contribution of approx. 0.36 kg for dissolved organic fragments. The dry mass of the IEX at the start including its loading is about 6.75 kg and about 8.9 kg considering additionally the contribution of the catalysts. By using an organic iron catalyst, the dry residue could be reduced by about 3 kg.

Further optimization such as recovery of the iron catalyst, low need for pH adjustment due to well-chosen catalysts or recycling and retreatment of organic fragments should improve the mass reduction factor to 2 (dry IEX with loading vs dry residue after liquefaction).

The liquefaction process for one batch lasts about 6 hours. During this time the energy demand is as follows: About 5.5 kWh are needed for the heating of the reaction solution in the pilot plant considering 20 liters IEX in 60 liters of aqueous medium. This energy demand is calculated from the heat capacities for water as well as for the pilot plant material steel (about 1 ton).

During the first 2 ½ h of the process (the most intensive cooling phase) 2.1 K (weighted) in average was discharged at an average coolant rate of 7.4 m<sup>3</sup>/h. During the second half of the process (again about 2 ½ h), 10.1 K (weighted) in average was discharged at an average cooling rate of 1.6 m<sup>3</sup>/h. This corresponds to a total cooling demand of 90 kWh during the process. For actively cooling down the reaction solution after liquefaction for further processing a cooling demand of 7.8 kWh was estimated to be required.

After the liquefaction process, the resulting liquid solution was concentrated using a rotating evaporator. The 235 liters of liquefied solution were concentrated to a volume of about 11 liters so that a volume reduction factor of 2 was achieved comparing the concentrated liquefied IEX with the wet IEX before processing.

### 3 CEMENTATION OF THE LIQUEFIED IEX

The development of appropriate cement formulations began with a baseline recipe derived from previous tests conducted at Framatome, containing cement, bentonite, superplasticizer and hydrated lime. The water required for the cementation was contained in the concentrated, liquefied IEX. Building on this initial formula, 18 new variations were tested by adjusting key parameters. The focus was put on enhancing the composition of the formulations to ensure volume optimization by maximizing waste incorporation and alignment with international waste acceptance criteria.

The final cement samples shown in Figure 3 had volumes similar to the initial corresponding IEX volume, which is about a factor 4 lower than the final volume resulting from direct cementation of spent IEX. The different cement recipe samples were subjected to compression strength test and temperature variation resistance test.

The majority of the samples passed these tests (90% for the compression strength, and 100 % for the temperature variation resistance) and 14 out of 18 cement variations passed the acceptance criteria of the tests resulting in a solidified waste product, which is suitable for final repository.



Figure 3: Photograph of the prepared cement samples made with concentrated liquefied IEX

For the successful cement recipes, a concentration of about 35 %-wt. of concentrated liquefied IEX was incorporated into the cement matrix. This corresponds to a concentration of 50 %-vol. of the concentrated liquefied IEX inside the cement matrix. Considering the volume reduction factor of 2 between the concentrates liquefied IEX and the untreated IEX, this results in a cemented waste product which has the same volume as the IEX prior to treatment. The final recipe for the cement matrix has to be adapted to the actual IEX type and loading coming from the client. Therefore, the actual composition of the solidified product is not stated in this paper.

### 4 DRYING OF THE LIQUEFIED IEX

For evaporator concentrates, drying is an alternative conditioning method to solidification via cementation. By concentration of the liquefied IEX inside the evaporator, concentrates are produced which are comparable in terms of their material properties (density, solid content, viscosity) to evaporator concentrates from a radioactive wastewater treatment of an NPP. The liquefied IEX have therefore been dried in a rotating drum drier system forming a solid powder after drying. The test criteria for a successful test were:

- Residual moisture < 10 %,
- Bulk Density of Dry Powder  $\geq 0.7 \text{ g/cm}^3$ ,
- No solid material remains sticking on the rotating drums of the rotating drum drier.

Several tests were performed with liquefied IEX and by mixing the liquefied IEX with other waste sources like evaporator concentrate simulates and boric acid. The resulting powder samples can be seen in Figure 4.

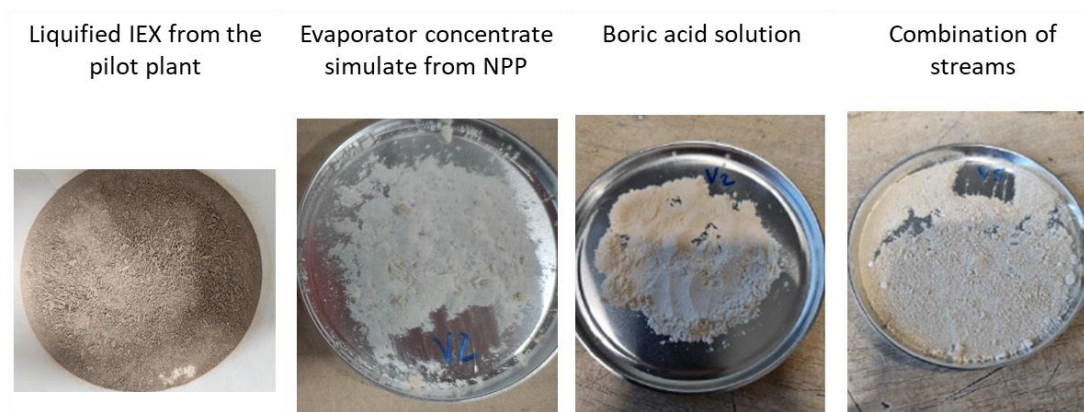


Figure 4: Resulting dried products from the rotating drum drier tests

All tests achieved the required test criteria. In total the volume reduction by drying of liquefied IEX with the rotating drum drier resulted in a factor of 5.6 to 9.4 in comparison with the volume of untreated resins.

## 5 GEOPOLYMERIZATION OF THE LIQUEFIED IEX

An emerging process to solidify radioactive material is geopolymerization in which the radioactive material is embedded into a geopolymer matrix. The geopolymer is an inorganic polymer-matrix on a silicate basis forming a covalently bonded polymer network thus creating a stable solid product. The benefits of geopolymers are:

- High water resistance,
- High fire resistance,
- High compressive strength,
- Purely mineral product without organic additives,
- Quick curing,
- Low CO<sub>2</sub> footprint.

Since the influence of the liquefied IEX on the geopolymer matrix is not known, it was decided to start with lower content of the concentrated liquefied IEX and increase the content step by step until the resulting geopolymer did not pass the acceptance criteria anymore. In addition, the inclusion of the dried powder from the tests with the rotating drum drier was tested as well, to see, if the total volume of embedded IEX could be increased this way. Differing from the development of cement recipes the development of geopolymer formulations is more complex. The different ingredients need to be adjusted to each other. The mixing order and time have also an impact. In

general, a geopolymer-matrix is formed with water glass and metakaolin as basis plus filler material to enhance the properties.

For the testing of the different geopolymer recipes, different samples were produced (as shown in Figure 5) and subjected to water resistance tests (by submerging the sample into a water bath) and compressive strength tests.

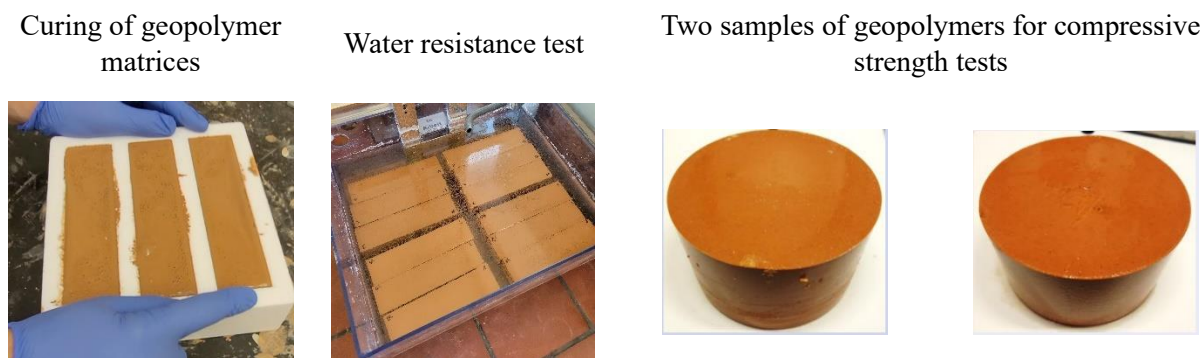


Figure 5: Photographs of the prepared geopolymer samples

The objective was to maximize the concentrated liquefied IEX or the dried liquefied content. The highest content of concentrated liquefied IEX achieved was about 20 %-wt. which still yielded compressive strength of about 10 N/mm<sup>2</sup>, the highest amount of dried liquefied IEX achieved was about 9 %-wt. with a compressive strength of about 10 N/mm<sup>2</sup>.

The corresponding concentration of untreated IEX would be:

- 40 %-wt. for the concentrated liquefied IEX and
- 66 %-wt. for the dried liquefied IEX.

A final recipe for the geopolymer matrix has to be adapted to the actual IEX type and loading coming from the client. Therefore, the actual composition of the solidified product is not stated in this paper.

## 6 CONCLUSIONS

Framatome has successfully commissioned and tested the Waste ERASER pilot plant for the decomposition of spent ion exchangers and demonstrated technical readiness. It has been shown that the system can be operated safely under mild conditions. The process developed in the laboratory has been further optimized regarding dosing concept and used catalysts.

Different conditioning processes for solidification of the liquefied IEX have been tested successfully, enabling different waste treatment paths for this type of waste product. Depending on the requirements of the final repository site and possible restrictions on the utilization of different processes this allows for a very flexible conditioning of spent resins.

In addition to the conditioning of the spent IEX themselves by drying or solidification, three further waste treatment routes are available resulting in final waste packages that have advantages, such as a reduced total volume of the packages and a long-term stability of the solidified product while still achieving similar results for the waste acceptance criteria like compressive strength. The different waste treatment paths are shown in Figure 6.

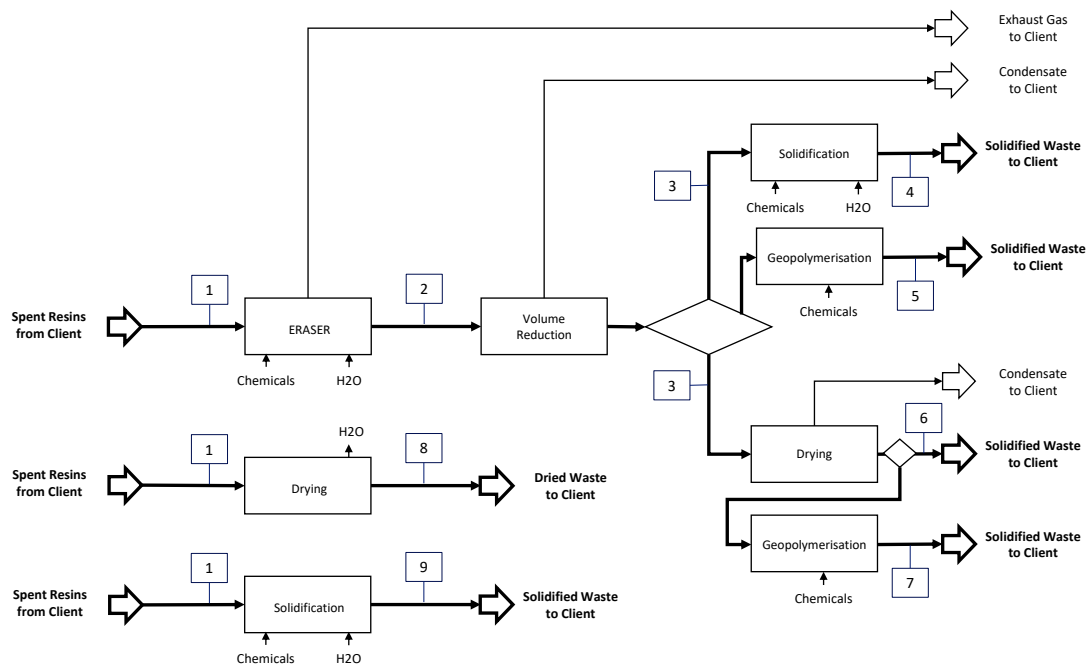


Figure 6: Possible waste treatment paths for IEX considering the Waste ERASER

These different conditioning processes for solidification of the liquefied IEX result in different waste volumes, which are collected in Table 1.

Table 1: Waste Volumes in relation to the initial Spent Resin Volumes for the different treatment steps according to Figure 6

| Step | Process                          | Relative Volume | Remark  |
|------|----------------------------------|-----------------|---|
| 1    | Spent Resins from Client         | 100%            | Requires conditioning                                 |
| 2    | ERASER                           | 800%            | Eliminates resin interactions, further treatment req. |
| 3    | Volume Reduction                 | 50%             | Product similar to evaporator concentrates            |
| 4    | Solidification                   | 100%            | Solidifying by incorporation into cement matrix       |
| 5    | Geopolimerization                | 167%            | Alternative solid matrix to cementation               |
| 6    | Drying                           | 18%             | High decrease of waste volume                         |
| 7    | Geopolimerization (after Drying) | 76%             | Further decrease of volume compared to step (5)       |
| 8    | Direct Drying                    | 50%             | Risk of swelling through re-hydration                 |
| 9    | Direct Solidification            | 300%            | Volume increase, risk of resin-cement interaction     |

In addition to the information collected in Table 1 the specific benefits and drawbacks for selected treatment steps are collected in Table 2.

Table 2: Benefits and drawbacks of selected conditioning methods

| <b>Criterion</b>                      | <b>Direct Solidification (9)</b>                        | <b>Solidification after ERASER (4)</b>   | <b>Geopolymerization after ERASER (5)</b>                                      | <b>Drying after ERASER (6)</b>  |
|---------------------------------------|---|--|--|---|
| Waste volume increase by conditioning | Factor of 3   | No increase, slightly decrease (Factor of 0.8-1)                               | Factor of 1.5 to 2.5   | No increase, high decrease (Factor of 0.18)                                 |
| Long-term pressure stability          | Low due to swelling and gas generation by decomposition | Excellent due to no swelling and no generation of gas by further decomposition | Excellent due to no swelling and no generation of gas by further decomposition | No direct pressure stability (powder), achieved by additional encapsulation |
| Specific activity of waste package    | Low, due to underloading                                | Medium   | Medium   | High  |
| Waste homogeneity                     | Low, Due to resin clusters                              | High, Full mixing of liquid solution with cement                               | High, Full mixing of liquid solution with geopolymer                           | High, Dried product is the final waste form                                 |

The tests with the liquefied IEX have shown, that the inclusion of the Waste ERASER process into the spent resin conditioning chain eliminates the main drawbacks of other conditioning methods and improves the overall process by minimizing the volume of the waste packages and improving the general formulation of a solidified waste product.

## ACKNOWLEDGEMENT

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## REFERENCES

- [1] Klaus, Buettner, Dr. Robert Lehr, Sven Nothvogel, Celine Boulet: “Chemical Treatment of Spent Ion Exchange Resin – 23058” – WMS 2023 Conference, February 26 – March 2, 2023, Phoenix, Arizona, USA
- [2] Klaus Buettner, Dr. Robert Lehr, Dr. Ines Beinert-Mertens, Thomas Fishedick, Wolfgang Schuch, Celine Boulet: “Successful Start of the Liquefaction Project for Spent Resin Treatment – 24367” - WM2024 Conference, March 10 – 14, 2024, Phoenix, Arizona, USA
- [3] Céline Boulet, Wolfgang Schuch, Gabriel Fuitem Martins: “Second Phase of the Project for Spent Resin Treatment: Solidification of the Liquefied Ion Exchange Resins– 25157” - WM2025 Conference, March 9 - 13, 2025, Phoenix, Arizona, USA
- [4] Céline Boulet, Wolfgang Schuch: “Rotating Drum Drier for Drying of Liquid Waste Including Liquefied Ion Exchange Resins, Evaporator Concentrate and Boric Acid– 25158” - WM2025 Conference, March 9 - 13, 2025, Phoenix, Arizona, USA