

Molten Salt Oxidation of Ion Exchange and Oils in Carbonate Salts and Evaporator Residue – 16531

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ABSTRACT

Molten Salt Oxidation is a well-studied and robust technology. Recently, renewed interest in the technology has been seen due to limitations on the use of incineration for hazardous and radioactive waste streams. The nuclear research center at Rez, near Prague, has been investigating a bench scale molten salt oxidation system to provide insight for the design of a pilot scale system to condition spent ion exchange resin and scintillator oils. The use of both an eutectic K/Na/Li carbonate salt and a simulated residue salt for liquid waste obtained from evaporated primary coolant of nuclear reactors was examined.

In these studies the usable capacity of the salts were examined. Materials doped with surrogate radioisotopes were processed and the capture efficiency was measured. Corrosion of the reactor vessel is of significant engineering concern. Corrosion coupon tests were performed for Inconel HX and stainless steel. These tests provided corrosion rates to assist in design of pilot scale systems.

While the bench scale reactor's detention time was too short to provide complete combustion, the values of emissions were tracked and showed an increase in the combustion efficiency at temperatures greater than 750 °C. A two part reactor has been designed and work is planned to continue to optimize the system.

Additionally, further work has examined the final conditioning of salt which has been saturated with radionuclides after processing organic waste streams. A conceptual pilot plant has been designed to solidify radionuclide saturated salt into a form acceptable for repositories according to EU directives. Saturated salt is milled to a powder and immobilized in a matrix with a high percentage of waste loading. Both geopolymer and polysiloxane matrices have been methods for low temperature immobilization. Conditioning in this manner decreases operating and capital costs when compared to current conditioning technologies and procedures utilized for high volume reduction factors of final waste forms.

INTRODUCTION AND BACKGROUND

Organic wastes are problematic to dispose of in underground repositories due to their potential for degradation and possible release of radionuclides for transport. Two of these waste types are contaminated liquid organic wastes (oils, scintillator liquids, solvents) and ion exchange (IX) resins. IX resins are polymer based beads with an active group that binds cations or anions. The resins are widely used in nuclear industry for several purposes, including cleaning primary circuit coolant. In some countries around the world, this waste has accumulated.

In the Dukovany Nuclear Power Plant (Czech Republic), IX resin waste has accumulated over decades of its operation. As of 2013, approximately 360 m³ of spent IX resins were stored here. [1] Recently, geopolymer matrices SIAL® and ALUSIL® were developed for efficient processing IX resins. However, this matrix is disadvantageous as it increases the final wasteform volume by at least two and a half times. This is ineffective considering high investment costs of repository and its limited storage/disposal volume. Therefore other technological solutions are desired.

Technologies with high volume reduction factors are desired. For organic wastes, one common technology is incineration. Incineration is industrially operated, however due to political, regulatory or social constraints it is not operated in other countries. A promising alternative thermal technology with a high volume reduction factor is Molten Salt Oxidation (MSO).

MSO is a non-flame catalytic liquid-phase oxidation process. [2] MSO is advantageous because it is operated at temperatures which are hundreds of degrees less than incineration. This is an important benefit since some radionuclides are volatile at incineration temperatures. Since MSO operates below the boiling temperature of these radionuclides and provides a safer treatment process. [2] Further, MSO does not generally require additional fuel, thus it generates a lower volume of off-gases which need treatment prior to release. In addition, the off-gas treatment system is simplified by the fact that the molten salt scrubs and captures acidic off-gases. Additionally, it is reported that permitting process for the MSO should be easier than incinerator permits due to the growing public opposition to incinerators. [3,4]

Molten Salt Oxidation is a well-developed, mature technology. [5,6] It is simple, consisting of a reactor vessel containing a bed of liquid molten salt, an oxidizing sparging system, and off-gas treatment. A solid, liquid or gaseous waste stream is fed below the surface of the molten salt bed combined with an oxidizing agent (generally air). Operation temperatures and salt compositions are varied depending on the salts used and according to the needs of temperature for oxidation of

organics processed. In the first applications of the MSO, Na_2CO_3 was used as a salt operating at nearly 1000°C . Since, for total destruction of many organic waste materials lower temperatures are sufficient. Different salts or salts mixtures (eutectics) can be used in the range of 200°C (AlCl_3) up to 1000°C (Na_2CO_3). For operating temperature around 600°C , an eutectic mixture of K, Li, Na carbonates were used. A good overview of tested salts is shown in literature. [7]

MSO was first investigated in the 1950s by Atomic International, a Division of North American Rockwell Corporation. [8] The technology was initially developed for removal of sulfur dioxide from power plant stack gases and then investigated as a catalyst for coal gasification. In the mid-1970s work started on investigating the potential to use MSO for treatment of hazardous and radioactive waste streams. Over the past 40 years, extensive research has been done at several research institutes in the US (LLNL, Oak Ridge, Indian Head Naval Surface Warfare Center, Engineered Technologies Energy Corporation), where the efficiency of the MSO for destruction of propellants, liquid wastes and solvents was studied. Most of the initial experiments were performed using bench scale (capacity of 0.45 – 4.5 kg/h) or pilot plant (capacity of 45 - 900 kg/h) instrumentation. In 1993 technical review report of MSO for DOE [9], the Panel summarizes according to the DOE technical papers based on the DOE funded research that “it supports the continuation of development of MSO, however assurance must be provided that outstanding technical issues” (described in the report [9]) be addressed at bench scale before proceeding with Title I design. The report summarizes the main issues connected with operation of the MSO technology.

Work on MSO has been performed at the Czech Nuclear Research Institute, UJV Rez, to develop a small system to treat solid IX and liquid oil wastes. Laboratory scale experiments were conducted to get practical experience with MSO technology for a planned pilot plant. The details of laboratory scale experiments are discussed in this paper. Laboratory experiments focusing on ion exchange resins destruction in molten carbonate eutectic at different airflow rates were performed, and compared with efficiency of destruction in the molten mock-up concentrate that should simulate concentrate produced in the primary circuit wastewater treatment system.

MATERIALS AND METHODS

Bench–Scale MSO Apparatus

A schematic of the MSO apparatus is shown in Figure 1. A 12 cm ID by 35 cm tall stainless steel reactor was constructed from 304 stainless steel. For most

experiments an additional 10 cm ID by 30 cm stainless steel crucible sleeve was utilized to minimize corrosion. For studies examining simulated evaporator residue an inconel crucible sleeve was utilized. A 0.2 cm ID Inconel 550 thermocouple well and 1 cm ID Inconel 550 downcomer were installed on a stainless steel flanged cover which was secured to the stainless steel reactor to form an airtight seal. IX resin was introduced through the use of a sealed hopper and screw feeder assembly. Oil was transmitted through the use of a pump. Compressed air was utilized both to introduce the simulated waste material to the reactor bed and provide the oxidant for reaction. Reaction gases exited the system through a vent at the top of the reactor lid, passed through a single tube heat exchanger, and were scrubbed through a borosilicate gas washing bottle filled with water before exiting to a fume hood. A portion of the exhaust gas was run through online gas analyzers and NO_x , CO , and O_2 were measured. A headspace of 18 cm between the top of the reactor and the surface of the salt was a function of the reactor design. The reactor was installed in a Clasic 2000 W electric furnace.

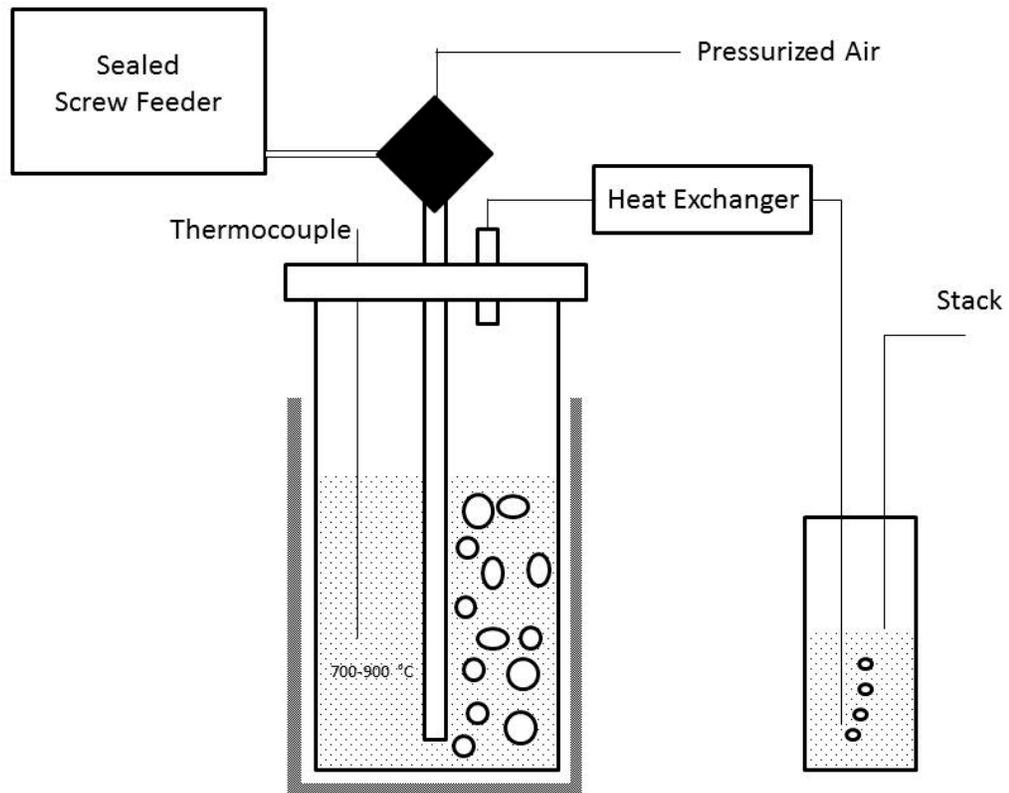


Fig. 1. Four liter reaction MSO apparatus.

Salt Composition

Two salt mixtures were evaluated for use in the MSO system. An eutectic carbonate salt was prepared as a mixture of 59mol% K_2CO_3 , 33mol% Na_2CO_3 , and 8mol% Li_2CO_3 . The eutectic melting temperature of the mixture is reported to be 401°C. [10,11,12]

A simulated evaporator residue was also evaluated. Fuel pool cooling water is evaporated to reduce volume leaving a residual salt which is difficult to process. This residual salt can be as high as 29×10^9 Bq/L and already must be disposed of within a repository. An investigation into the use of this salt as the molten medium for MSO was undertaken. By utilizing a waste stream as the molten medium, and concentrating radionuclides contained on organic wastes, the effective volume of the organic waste stream would be significantly reduced. Table 1 shows provides the composition the simulated evaporator residue.

Table 1: Composition of simulated evaporator residue.

Component	wt%
H_3BO_3	33.62
NaCl	1.68
Na_2CO_3	21.21
Na_2NO_3	12.34
Na_2SO_3	2.3
$Na(COO)_2$	2.53
$NaNO_2$	1.42
KNO_2	10.45
NaOH	14.41

Simulated Wastes

Both IX resins and waste oils were examined. Purolite A-400 anionic exchange resin and C-100H cationic exchange resin were used. Both have a polystyrene polymer structure crosslinked with divinylbenzene and have functional groups of R_4N^+ and SO_3-H^+ respectively. Resin was loaded with a solution of strontium or cobalt to serve as a non-radioactive surrogate for Sr-90 or Co-60. Ash and moisture content of the resin was measured to be 17.0% and 22% respectively. The resin was dried at 60°C before introduction to the MSO reactor. A heating exchange grade oil was utilized to simulate waste scintillator oils. Contamination was simulated by creating a cobalt, strontium, or cesium spiked oil/water emulsion to simulate Co-60, Sr-90, Cs-137 contamination.

Analysis

To measure the effectiveness of MSO as a treatment technology, destruction efficiency was tracked through both solid and gaseous samples. Online gas analyzers were connected to the system to track combustion efficiency and determine what emission controls may be needed for operations at scale. Initially a Hartmann & Braun Advance Optima Uras-14 was used to measure NO_x and CO and a Siemens OxyMat 5M was utilized to measure O₂. The gas was filtered through a prefilter to remove any long chain hydrocarbons. Additional samples were analyzed by gas chromatography to measure CO, CO₂, NO_x, and total hydrocarbons.

Samples of the salt mixture were taken throughout the experiments. Metal ion content was tracked by in the salt by ICP-MS to determine the entrapment efficiency of surrogate radioactive metal entrainment. X-Ray Diffraction (XRD) was also performed to determine the speciation of surrogate radioactive complexes.

Corrosion

Corrosion of materials in elevated salt environments is of significant concern. [13] In a report, Rockwell International suggested that stainless steel 347 would be a suitable material of construction after performing extensive testing. [8] Another study by the Solar Energy Research Institute showed Inconel 600 to be the ideal material of construction. [14] This literature led to the choice of using stainless steel for the reactor vessel and inconel for the downcomer as it appeared the corrosion rates were slow enough to allow for a bench scale apparatus.

However, experimentally, both inconel and stainless steel saw corrosion in both the eutectic carbonate salt and the simulated evaporator residue. Due to these observations, studies were performed by Sulejmanov, 2015, to determine a rate and mechanism of corrosion. These studies were performed to determine engineering parameters for a planned semi-scale MSO pilot plant. Studies were performed by submerged coupon tests for up to 96 hours. Analysis was performed by both surface and cut sample microscopy on an Olympus BX51 microscope. Additionally, weight loss measurements were taken at intervals. [15]

Operating Conditions

Utilizing a molten eutectic carbonate salt, conditions within the MSO reactor can be modeled as a bubble column reactor. [16] Even though the density and viscosity of the salt are higher than water at elevated temperatures, the behavior of the system can be approximated to that of an aqueous system. [17] The minimum detention time of a particle in the molten salt bed is equal to the gas retention time. Utilizing temperature corrected values for salt density and viscosity obtained from Janz 1979, the gaseous residence time was calculated to be between 1 and 1.8 seconds. [16,17,18] Operational temperatures were varied from 650-900 °C. The

stoichiometric air to fuel ratio was varied from $\lambda=1.1$ to $\lambda=1.9$. The stoichiometric air to fuel ratio is a description of the stoichiometric oxidant requirements for the fuel. A ratio of $\lambda=1.0$ describes the molar equivalent for complete combustion while $\lambda>1.0$ is a lean mixture and $\lambda<1.0$ is a rich mixture. An optimum operation temperature was chosen at 750 °C with $\lambda=1.5$. [19]

When using the simulated evaporator residue, the viscosity of the salt was much higher. Operating conditions of 850 °C – 950 °C were used to provide better reaction kinetics. The air ratio of $\lambda=1.5$ was maintained.

RESULTS AND DISCUSSION

Destruction Efficiency

The overall efficiency of the MSO process for the destruction of ion exchange resins is determined by evaluating the overall volume reduction and the retention of radioactive surrogate ions within the salt matrix.

Polystyrene resins are reported to decompose in three stages. Dehydration occurs between 90-200 °C, disassociation of the functional groups and the depolymerization of styrene-divinylbenzene structures occurs between 380-440 °C, and complete oxidation occurs between 500-1000 °C. [20] Table I compares carbon content in samples of resin “as received” from the manufacturer, ash taken after incineration at 800 °C, and the insoluble ash obtained after dissolution of salt from the MSO reactor. Oxidation within the MSO reactor is improved over incineration techniques due to increased contact and agitation.

Table 2: Carbon analysis of Resin and Ash

Carbon Analysis	As Received Resin ¹	Incinerator Ash	MSO Ash
	Wt. %	Wt. %	Wt. %
Organic	1.9	5.3	6.4
Inorganic	29.3	33.7	<6.4

The values given in the table above shows that in “as received resin”, there is more insoluble carbon, which is represented by stable aromatic molecules of styrene. This carbon form is not decomposed by the chromsulfuric acid as would be the case for simpler aliphatic compounds. After flameless heating at the same temperature as present in the molten salt, some part of the styrene decomposes into aliphatic

¹ The “as received” moisture content of Purolite C100H was 22%.

hydrocarbon chains. The resin forms a clinker particle and the physical structure (shape) remains in bead form. The color is changed from orange to gray. After application of MSO, the insoluble styrene organic form was not detected. In an experiment under non optimum stoichiometric combustion ($\lambda=0.75$), the salt was found to retain particles as a clinker. These particles were oxidized, without the addition of further resin, and observed to fully combust in approximately three hours. This result gives a qualitative detention time of the resin. Figure 2 shows the progression of this experiment using qualitative grab samples.



Figure 2: Qualitative experimental results from non-stoichiometric oxidation experiments and subsequent oxidation. Carbon clinker in the melt was observed to oxidize fully after 3 hours.

Off-Gas Emissions

Off-gases from the reactor were analyzed through online analyzers. The off-gas showed elevated levels of CO and NO_x , exceeding emission requirements for both US EPA and Czech Republic regulations. Steady state emissions of NO_x were measured between 78-130 ppmv. Emissions of CO are a function of temperature and the stoichiometric air to fuel ratio. As shown in Figure 3, as temperature increases CO emissions decrease. Higher stoichiometric ratio conditions ($\lambda > 1.5$) increase CO due to more efficient oxidation. Pyrolytic formation of hydrocarbons was seen at stoichiometric ratios below one ($\lambda = 0.75$) creating unstable reactor conditions. Figure 4 describes the oxygen conditions throughout the experimental work.

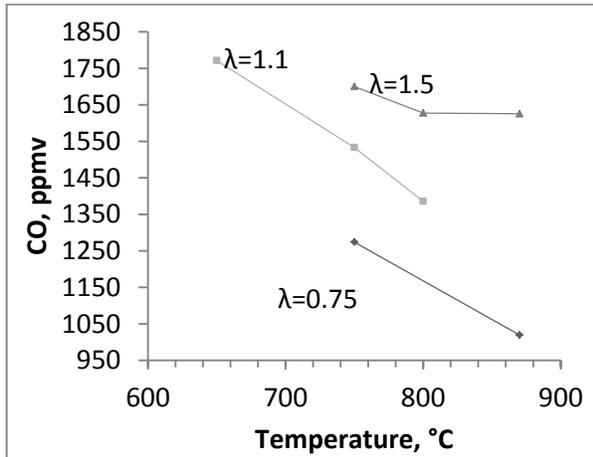


Fig. 3. Averaged CO emissions from MSO

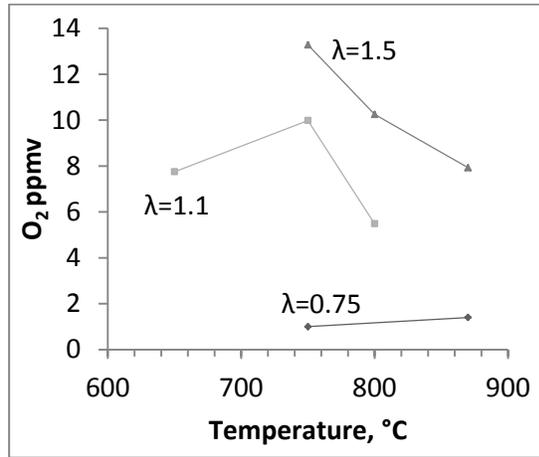


Fig. 4. Averaged O₂ excess from MSO

Salt Life

For the MSO system to be a useful technology, the amount of resin which can be processed must be greater than the volume of waste salt which is produced. Chemical analysis confirmed the retention of surrogate radionuclides within the salt. XRD also indicated the presence of complexed radionuclide carbonate compounds in the eutectic carbonate system. In experiments no noticeable changes in viscosity have been found, though literature has reported a maximum ash content of 20 – 50 wt.% as the limiting factor in processing solid waste streams. Work is currently being performed to determine the volume of waste which may be processed. Particulate ash entrainment from these preliminary results can be seen in Figure 5.

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Fig. 5. Comparison ash entrained in grab samples

Corrosion

Corrosion of the reactor vessel is of significant design concern for full scale applications. Corrosion within molten carbonate systems has been widely studied. [21,22] Hot corrosion is reported to deplete chromium and form an oxide surface layer. This is reported to be especially pronounced in the presence of sulfur and chloride compounds. [23]

Surface oxidation appears to have formed on both stainless steels and inconel alloys during bench scale studies with eutectic carbonate salts. This oxide layer was seen violently cracking and flaking off during periods of thermal cycling. This appears to indicate that the oxide layer fails cohesively with tensile stress gradients which form in the oxide layer upon rapid cooling. [24] Further investigation will need to be conducted to understand the corrosion fully prior to pilot plant implementations.

While performing preliminary bench scale studies using evaporator residue, extreme corrosion was seen. In some cases, the corrosion was seen within a matter of hours. Since no studies to characterize the corrosiveness of molten evaporator residue existed, a study was performed by Sulejmanov, 2015. [15] This study showed that the evaporator residue was significantly more corrosive than eutectic carbonates on stainless steel and slightly more corrosive on inconel. At 900 °C, corrosion rates were observed to be 11.4 – 15.1 mm/yr for Inconel 713 and 120 – 381mm/yr for ASTM 316Ti stainless steel. Experimental results can be seen in Tables 3 and 4.

Surface morphology and cross section morphology was performed on the samples. These analyses show that a surface oxide forms and pitting starts to occur on both Inconel 713 and ASTM 316Ti. Sulejmanov theorized that chromium depletion is the corrosion mechanism, in a similar manner as in molten carbonate salts. Figures 6 and 7 show cross section morphologies of Inconel 713. Figures 8 and 9 show morphologies of ASTM 316Ti. [15]

Table 3: Submersion tests for Inconel 713 coupons in molten simulated evaporator residue salt at 900 °C

Inconel 713						
Time, h	A, cm ²	ρ, g/cm ³	m initial, g	m final, g	Δm, g	Corrosion Rate, mm/yr
24	4.39	7.93	1.4964	1.3521	0.1443	15.1 (±) 0.7
48	4.45	7.93	1.5113	1.2245	0.2868	14.8 (±) 0.6
96	4.35	7.93	1.5091	1.0793	0.4298	11.4 (±) 0.2
192	4.47	7.93	1.6014	Destroyed	-	-

Table 4: Submersion tests for stainless steel ASTM316Ti coupons in molten simulated evaporator residue salt at 900 °C

Stainless Steel ASTM 316Ti						
Time, h	A, cm ²	ρ , g/cm ³	m initial, g	m final, g	Δ m, g	Corrosion Rate, mm/yr
1	2.87	7.99	1.4036	1.3721	0.0315	120 (\pm) 13
2	2.87	7.99	1.4275	1.2430	0.1845	352 (\pm) 18
4	2.81	7.99	1.2910	0.9005	0.3905	381 (\pm) 19
8	5.62	7.99	2.9578	1.5898	1.3680	333 (\pm) 17
16	2.88	7.99	1.3367	0.0820	1.2547	298 (\pm) 9
24	2.95	7.99	1.4300	Destroyed	-	-
48	2.77	7.99	1.2329	Destroyed	-	-
96	2.82	7.99	1.2728	Destroyed	-	-
192	2.97	7.99	1.4455	Destroyed	-	-

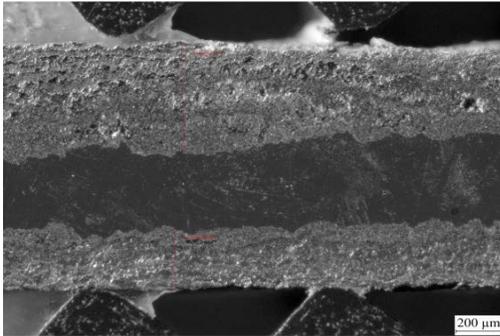


Figure 6

Figure 6: Cross section view of Inconel 713 after 96h at 200 μ m

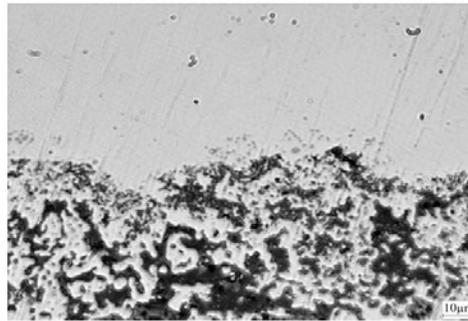


Figure 7

Figure 7: Cross section view of Inconel 713 after 96h at 10 μ m

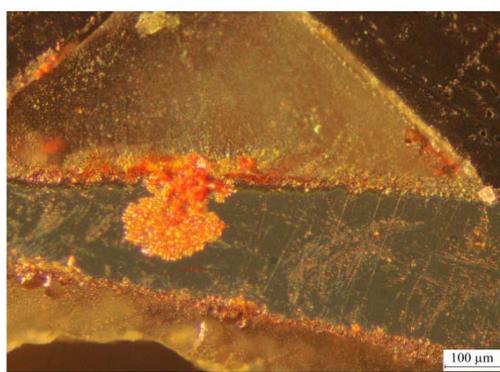


Figure 8

Figure 8: Cross section view of Stainless Steel ASTM 316Ti after 16 hours at 100 μ m

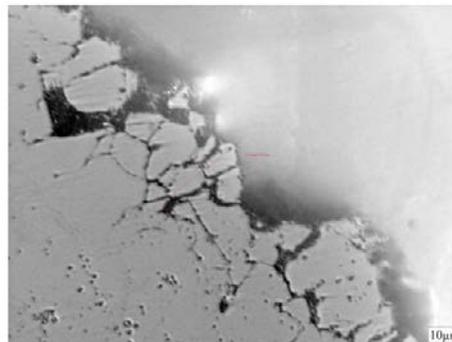


Figure 9

Figure 9: Cross section view of Stainless Steel ASTM 316Ti after 4 hours at 10 μ m

DISCUSSION

The bench-scale apparatus for MSO technology verification was put in operation in Research Centre Rez, the Czech Republic, with the aim to perform preliminary experiments to gain knowledge for design and construction of pilot-plant MSO unit.

In the Czech Republic, over 300 m³ of spent IX resins has accumulated. These IX resins are difficult to treat by available technologies. The bench scale MSO apparatus demonstrated that this technology is suitable for the destruction of IX resins and waste organic liquids.

Simulated organic wastes containing surrogates for Co-60, Sr-90, Cs-137 were processed and the destruction efficiency was measured. In these preliminary tests, MSO captured simulated radionuclides. Further, the organic waste stream was effectively destroyed through low temperature flameless oxidation. The volume of the organic waste stream is effectively reduced, and an inorganic waste can be conditioned with a geopolymer matrix for disposal. This significantly reduces the overall volume of the waste which must be disposed in a repository.

However several issues that must be solved before its application were found. Air emissions were above regulatory limits and control systems will need to be installed on the pilot scale system. These emission controls should be a function of both reactor design as well as engineering controls.

Corrosion is of significant engineering concern. Corrosion rates for Inconel 713 and stainless steel ASTM316Ti were examined for the simulated evaporator salt system. The study showed extreme corrosion in stainless steel and a high corrosion rate to inconel. Specific coatings or engineering solutions will need to be developed to combat this corrosion.

MSO has some technical challenges, however it has great promise to provide a tool for waste volume reduction of characterized organic waste streams such as oils or ion exchange resins.

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