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Separation of Cesium and Strontium from Residues Arising from Fluoride Volatility Processing of Spent Nuclear Fuel

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Abstract: The overall objective of this study was to support an alternative hybrid process to meet Advanced Fuel Cycle Initiative goals, using fluorination and aqueous processing techniques, for treatment of spent nuclear fuel (SNF). The specific goal was to develop a simple aqueous dissolution process to separate two high-heat fission products, cesium and strontium, from SNF fluoride residues. This separation was based on solubility differences examined by modeling using the HSC Chemistry 5.0 program. HSC automatically utilizes an extensive thermochemical database, which contains enthalpy (H), entropy (S), and heat capacity (C) data for more than 17,000 chemical compounds. The work focused on the fluoride residues from the voloxidation and fluorination steps of the fluoride volatility process and was limited to SNF from commercial light-water reactors. Material balances were used to estimate the quantity of residue. A representative SNF was considered to be one with a burnup of 33,000 megawatt days per metric ton initial heavy metal after a 10-yr cooling period, from a pressurized-water reactor. The dry fluorination method was used for uranium removal. The work described in this paper is based solely on computer modeling, which will serve as the basis for any necessary follow-on laboratory validation experiments. Observations from this study showed that water dissolution provided adequate separation of cesium from the fluoride residues but only negligible separation of strontium with cesium. After removal of cesium, a second separation with water provided little additional removal of strontium. For disposal purposes, it would be reasonable to dispose of cesium and strontium together. Therefore, more research is needed to examine the possibility of converting all fluoride residues to hydroxides to increase the solubility of strontium.

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INTRODUCTION

Over the last 50 yrs, the principal reason for reprocessing has been to recover unused uranium and plutonium from spent nuclear fuel (SNF) elements. A secondary reason for reprocessing has been to reduce the amount of material to be disposed of as high-level waste and to create a more stable waste form. For environmental and economic reasons, it is desirable to reduce the number and volume of waste streams from nuclear fuel reprocessing operations (1). The development of advanced reprocessing technology should be planned to achieve economy, nonproliferation, and reduction of radioactive wastes at the same time (2). Currently in the United States, the motivation for reprocessing civilian SNF has been to extend the lifetime of the repository as a means to reduce the life-cycle costs of nuclear electricity production.

Recently, interest has grown in separating individual radionuclides from SNF reprocessing waste to (a) reduce long-term radiotoxicity in residual wastes, (b) support transmutation of long-lived radionuclides into shorter-lived or stable isotopes, and (c) improve repository heat management. The management of vitrified high-activity waste arising from the reprocessing of SNF is often made questionable by the existence of long-lived radionuclides, especially the minor actinides and certain fission products (3). The elimination of these radionuclides from commercial SNF intended for disposal in a mined repository can have a significant positive effect on the overall performance of the repository (4–5). The main radionuclides targeted for separation are the actinides: Np, Am, and Cm (along with U and Pu) and the fission products: I, Tc, Cs, and Sr. Removal of the latter two significantly reduces the heat load of the residual conditioned wastes. Strontium^{90} and ^{137}Cs, both short-half-life fission products, almost completely determine the total toxicity and heat generation of the fission product nuclides ($<$300 yrs) (6). High-activity wastes, which contain minor actinides (Np, Am, Cm, and higher actinides) and fission products (FP), arising from the reprocessing of spent fuels are currently being immobilized in a glass matrix for subsequent disposal in a deep underground repository (3).

In 2001, the total costs for a geological repository at Yucca Mountain for the disposal of SNF and high-level radioactive waste were estimated to be between $42.8 and $57.1 billion (7). Such a costly endeavor provides an incentive to more seriously consider techniques that would extend the life of the repository, if not completely avoid the need for additional repositories in the future. At current production rates and without reprocessing, the SNF inventory will reach the statutory capacity of the Yucca Mountain Repository before the year 2010 (8). Separation of SNF into various components offers a way to extend the life of the repository. For example, uranium could be removed to reduce the mass of SNF, and cesium and strontium could be removed to reduce the heat load on the repository. Additional products

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could also be recovered for use as fuels (U, Pu, and Np) or for transmutation (Am, Cm, I, and Tc).

The goals of the Advanced Fuel Cycle Initiative (AFCI) are outlined in the Report to Congress (9). The first goal of the AFCI is to process SNF in order to reduce the volume of waste requiring repository disposal. This can be accomplished by separation of the SNF into its main components. Spent fuel from commercial light-water reactors (LWRs) is approximately 95% uranium, 1% plutonium, and 4% FP. Separation would allow for the removal of uranium, which would greatly reduce the mass of SNF. Separation of cesium and strontium could be performed to further reduce the heat load on the repository. The second goal of the AFCI is to separate long-lived, highly toxic elements, such as plutonium, for mixed oxide (MOX) fuels and americium and curium for transmutation. A third goal is to reclaim the valuable energy reserves of SNF. This could be accomplished by reenriching the uranium removed and by using the recovered plutonium and neptunium in MOX fuels. Finally, the AFCI strives to accomplish the goals discussed above in a proliferation-resistant manner. This could be accomplished by coprocessing plutonium and neptunium, therefore avoiding separation of purified plutonium.

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Traditional reprocessing methods require high temperatures, acidic conditions, and organic solvents, which increase the volume of wastes that must be disposed of as low-level or intermediate wastes. The wastes contain phosphates from the solvents, which potentially limit the amount of radionuclides that can be vitrified. The contaminated solvents would be incinerated and then the ash would be vitrified. A simplified and less costly reprocessing technique is desired.

The overall objective of this work was to support an alternative hybrid process to meet the AFCI goals using fluorination and aqueous processing techniques for treatment of SNF. The specific goal was to develop a simple aqueous process for partitioning the residue from the fluorination stage of the fluoride volatility process. The work was designed to examine the separation of high-heat fission products from fluoride residues using simple dissolution methods. This separation was based on estimates of solubility differences predicted by computer simulations. The remainder of the residue could be considered waste or potentially further processed to recover Pu/Np and/or Am/Cm.

Conceptual process flow diagrams for the processing of SNF and the separation of fluoride residues are shown in Figs. 1 and 2, respectively. Figure 1 shows the treatment of SNF from disassembly through fluorination. The fuel rods are disassembled and decladded. The hardware goes to additional processing or disposal, while the SNF enters a voloxidator. In the voloxidator, air and oxygen gas are used to convert the components of the SNF to oxides. Volatile oxides are treated as needed, and nonvolatile oxides are spent to fluorination where fluorine gas is used. Fluoride volatiles, comprised mainly of UF_6, are

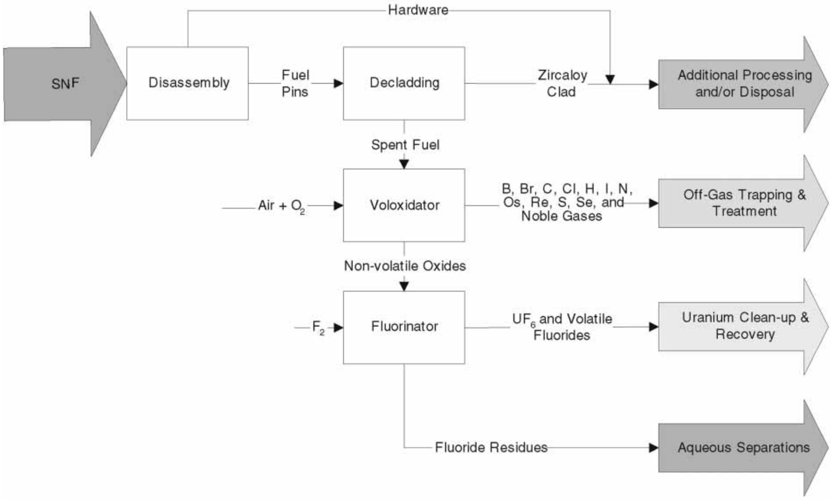

Figure 1. Flow diagram for processing of SNF, leading to aqueous separation of fluoride residues.

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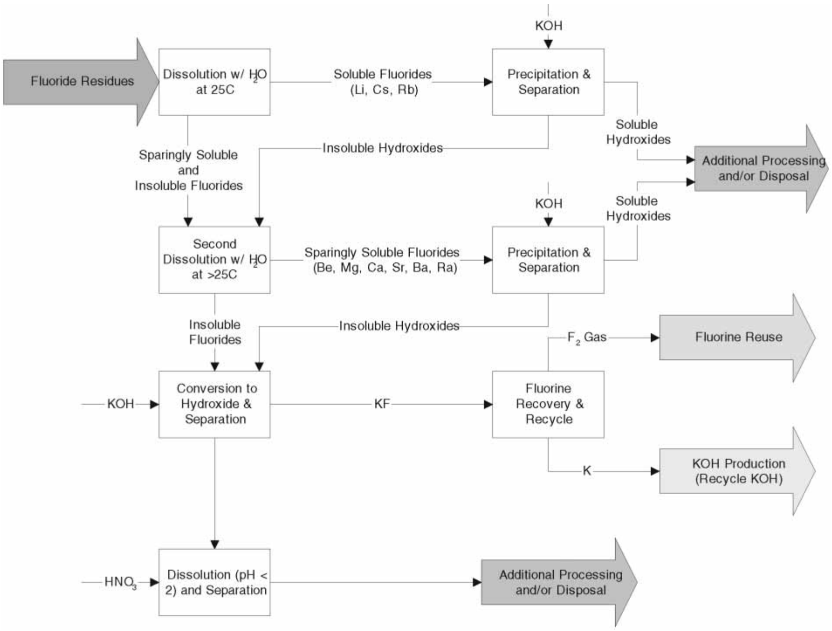

Figure 2. Concept of aqueous separation of fluoride residues.

sent to uranium clean-up and recovery. The nonvolatile fluoride residues are treated using aqueous separations. Figure 2 shows a conceptual process for the aqueous separation of fluoride residues. First, the residues are treated with water to remove soluble fluorides. These soluble fluorides are treated with KOH to remove impurities, such small amounts of rare earth fluorides that solublize. The soluble hydroxides are sent for additional processing as necessary. The insoluble fluorides are treated a second time with water, and the soluble fluorides from this treatment are also treated with KOH. The second water treatment is necessary due to the common ion effect of CsF and SrF_2 interactions. The highly soluble CsF causes SrF_2, a sparingly soluble species, to be less soluble. These soluble hydroxides are sent for additional processing, as needed. Potassium hydroxide is added to the insoluble fluorides. Potassium fluoride is released and sent to fluorine recovery and recycle. The fluorine gas is reused in the fluorinator, while the potassium is used in KOH production. Any remaining insoluble species are acidified with HNO_3 and then sent for additional processing and disposal.

The present work is a study of the selective dissolution of fluorides remaining from the fluoride volatility process. The work focused on the fluoride residues from the preceding voloxidation and fluorination steps of the fluoride volatility process and was limited to a representative SNF system from a pressurized-water reactor (PWR) with an average fuel burnup of 33,000 megawatt days per metric ton initial heavy metal (MWd/MTIHM) and a 10-yr cooling period. This particular fuel was selected as a reasonable representative fuel based on current inventories of SNF. Most commercial reactors are PWR, and the selected burnup is typical of these reactors. All spent fuel is cooled at least 5 yrs. The longer SNF is allowed to cool; the less radioactive material remains due to decay. The majority of SNF currently in inventory has been cooled for more than 10 yrs. Material balances were used to estimate the quantity of residue.

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METHOD

Definition of Chemical System

The HSC Chemistry 5.0 program (10) was used throughout this project. HSC was designed for various kinds of chemical reactions and equilibria calculations. The current version contains 14 calculation modules displayed as 14 options in the HSC main menu:

1. Reaction Equations
2. Heat and Material Balances

3. Heat Loss Calculations
4. Equilibrium Compositions
5. Electrochemical Equilibria
6. Formula Weights
7. Eh – pH – Diagrams
8. H, S, C, and G Diagrams
9. Phase Stability Diagrams
10. Mineralogy Iterations
11. Composition Conversions
12. Elements
13. Water (Steam Tables)
14. Units

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One feature of HSC was that all 14-calculation options automatically utilize the same extensive thermochemical database, which contains enthalpy (H), entropy (S), and heat capacity (C) data for more than 17,000 chemical compounds. HSC Chemistry offers calculation methods for studying the effects of different variables on the chemical system at equilibrium. However, HSC does not solve all chemical problems, because the kinetics of the chemical reactions and nonideality of solutions are not taken into account. It is possible to add the necessary information to the module. Modules 1, 4, and 9 were used almost exclusively in this research.

A representative SNF system from a PWR with an average fuel burnup of 33,000 MWd/MTIHM and cooled for 10 yrs was used. The assay of the elemental and radionuclide components of the SNF is detailed in a report by Croff (11). Negligible carryover of SNF in the cladding was assumed. In order to simplify the mathematical modeling, insoluble materials were not considered in the model.

Material balances were performed on the voloxidation and fluorination steps to determine which components would volatilize. Using the HSC Chemistry Phase Stability Diagrams module, phase stability diagrams were produced to determine which form of each component would predominate after fluorination. The remaining components were classified as primary or trace in order to simplify the initial modeling efforts. Primary components were defined as any element present in quantities greater than 0.01 wt% of the total mass, and trace components were defined as any element less than or equal to 0.01 wt % down to 10^{-10} g. Components initially present in SNF in quantities less than 10^{-10} g were considered zero. Table 1 lists the component concentrations of the volatile stream leaving the voloxidator. Table 2 lists the component concentrations of the volatile stream leaving the fluorinator, and Table 3 lists the component concentrations of the fluoride residue.

Table 1. Composition of off-gas stream leaving voloxidizer

Volatile oxides	mol %
Ar	4.36E − 05
Br2	0.70
CO2	30.99
Cl2	0.38
H2	0.10
He	1.03
I2	4.74
Kr	5.47
N2	4.54
Ne	2.41E − 05
OsO4	1.06E − 03
Re2O7	9.90E − 03
Rn	4.77E − 14
SO3	1.60E − 03
Xe	52.04

Modeling Approach

The HSC Chemistry Equilibrium Compositions Module was used to model the system and determine which components would dissolve and which would remain insoluble.

Table 2. Composition of volatile fluoride stream from fluorinator

Volatile fluorides	mol %
AsF3	3.75E − 05
BF3	1.07E − 03
GeF2	9.58E − 05
MoF6	0.86
PF5	2.38E − 02
SbF5	3.39E − 03
SeF4	1.25E − 02
SiF4	7.57E − 03
TcF6	0.19
Te2F10	0.16
UF6	98.74
VF5	1.23E − 03
WF6	2.42E − 04

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Table 3. Composition of fluoride residues leaving fluorinator

Primary species	mol %	Trace species	mol %
AgF2	0.209	AcF3	9.86E − 11
AlF3	0.243	BeF2	2.59E − 05
AmF4	1.170	BiF4	9.41E − 04
BaF2	3.702	CaF2	1.47E − 02
CdF2	0.348	Cf	4.93E − 11
CeF4	8.297	CoF3	6.81E − 03
CmO2	0.016	CuF2	4.69E − 03
CrF3	0.076	DyF3	3.38E − 03
CsF	3.524	ErF3	1.32E − 04
EuF3	0.341	GaF3	1.80E − 04
FeF3	0.141	HoF3	3.39E − 04
GdF3	0.035	InF3	8.89E − 03
LaF3	3.442	IrF5	2.01E − 09
NaF	0.128	KF	3.50E − 07
NdF3	10.973	LiF	3.04E − 02
NiF2	0.101	MgF2	2.47E − 02
NpF6	1.298	MnF3	1.15E − 02
PdF2	3.799	NbF5	2.82E − 05
Pm2O3	0.036	PaF5	9.06E − 07
PrF4	3.893	PbF4	2.37E − 03
PuF4	17.508	Pt	1.49E − 10
RbF	0.811	RaF2	4.90E − 10
RhF4	2.232	ScF3	8.83E − 07
RuF4	10.597	TaF5	9.78E − 07
SmF3	2.251	TbF3	6.48E − 03
SnF4	0.397	ThF4	1.62E − 05
SrF2	2.600	TiF4	1.03E − 02
YF3	2.018	TmF3	1.30E − 07
ZnF2	0.181	YbF3	4.07E − 08
ZrF4	19.505		

Testing of single-component systems with known literature values for solubility (12) was used to validate the HSC Chemistry Equilibrium model. For each component, 1 kmol of compound was added to 100 mL of water. The model returns the concentration as a function of temperature to indicate the solubility of each component.

The HSC Chemistry Equilibrium Compositions module was used to model the system response to an increase in temperature. The temperature was increased from 0 to 100°C in 5-degree increments, and 1 kmol of water was used. Table 4 lists the quantities of the expected components input for the calculation by the HSC Equilibrium Compositions Module. Solubility

Table 4. HSC chemistry 5.0 input for modeling of separation of Cs/Sr

Species	Input concentration (kmol)	Solubility (g/100 mL water@oC)
BaF2	1.13E − 03	0.12 (25)
BeF2	7.92E − 09	Soluble
CaF2	4.50E − 06	0.0016 (18), 0.0017 (26)
CdF2	1.07E − 04	4.35 (25)
CsF	1.08E − 03	367 (18)
InF3	2.72E − 06	0.040 (25)
KF	1.07E − 10	92.3 (18)
LiF	9.31E − 06	0.27 (18)
MgF2	7.55E − 06	33 (cold)
NaF	3.91E − 05	4.22 (18)
RaF2	1.50E − 13	—
RbF	2.48E − 04	130.6 (18)
SrF2	7.95E − 04	0.011 (0), 0.012 (27)
ZnF2	5.55E − 05	1.62 (20)
ZrF4	5.96E − 03	1.388 (25)

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data for each species are also listed in Table 4. For modeling the second dissolution with water, all species from Group 1 (alkali) of the periodic table were removed from the input list.

RESULTS AND DISCUSSION

The stability of the fluoride residues in water was determined using the HSC Reaction Equation module. In this module, the potential reaction equation was input along with a temperature range. The module returned the equilibrium constants for the specified temperature range. The stability of the fluoride residues was tested by modeling of the reaction of the fluoride species with water to produce HF. For the process studied, it was necessary to determine whether or not HF would be produced. Hydrogen fluoride production would make the separation of the fluoride residues difficult because many metal fluorides are soluble in aqueous HF. Potential problem reactions with equilibrium constants at 25°C are shown in Table 5.

The HSC Chemistry 5.0 model accurately predicted the qualitative solubility of each species tested. As a single component in water, CsF is very soluble in water (367 g/100 mL H_2O at 18°C); however, SrF_2 is only slightly soluble in water (0.012 g/100 mL H_2O at 27°C). Due to the common ion effect, CsF, as well as the other ions in solution, suppresses the solubility of SrF_2. HSC predicted the solubility of strontium in water

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Table 5. Potential HF-forming reactions

Species	Potential reaction	log K@25C
AgF2	AgF2 + H2O(l) = AgO + 2HF(a)	6.980
CoF3	CoF3 + 3H2O(l) = Co(OH)3 + 3HF(a)	9.890
FeF3	2FeF3(ia) + 3H2O(l) = Fe2O3 + 6HF(a)	22.455
	FeF3(ia) + 3H2O(l) = Fe(OH)3 + 3HF(a)	8.205
	FeF3(ia) + 2H2O(l) = Fe(OH)O(a) + 3HF(a)	3.020
NbF5	2NbF5 + 5H2O(l) = 10HF(a) + Nb2O5	26.280
	NbF5 + 2H2O(l) = 4HF(a) + NbO2F	8.820
	NbF5 + 3H2O(l) = 5HF(a) + Nb(OH)O2(a)	12.172
PbF4	PbF4 + 2H2O(l) = 4HF(a) + PbO2	16.511
PdF2	PdF2 + H2O(l) = PdO + 2HF(a)	3.222
PuF4	PuF4 + 2H2O(l) = PuO2 + 4HF(a)	4.581
RuF4	RuF4 + 2H2O(l) = RuO2 + 4HF(a)	15.175
SnF4	SnF4 + 2H2O(l) = SnO2 + 4HF(a)	26.144
	SnF4(ia) + 2H2O(l) = SnO2 + 4HF(a)	21.100
	SnF4 + 4H2O(l) = Sn(OH)4 + 4HF(a)	19.532
	SnF4 + 4H2O(l) = Sn(OH)4(a) + 4HF(a)	19.210
	SnF4(ia) + 4H2O(l) = Sn(OH)4 + 4HF(a)	14.488
	SnF4(ia) + 4H2O(l) = Sn(OH)4(a) + 4HF(a)	14.166
TaF5	2TaF5 + 5H2O(l) = Ta2O5 + 10HF(a)	19.729
	TaF5 + 2H2O(l) = TaFO2 + 4HF(a)	4.607
TiF4	TiF4 + 2H2O(l) = TiO2 + 4HF(a)	7.573
TmF3	2TmF3 + 3H2O(l) = 6HF(a) + Tm2O3	1.709

containing fluoride residues to be 0.00107 g/100 mL. Figure 3 shows the HSC Chemistry result for the separation of Cs/Sr from SNF via dissolution in water. The composition of each species determines the solubility. In Fig. 3, SrF_2 and Cs(+a) are the largest components. Strontium fluoride, as shown in the figure, was in the solid form; Cs(+a) was the aqueous form of the cesium ion. The first dissolution with water was effective for the removal of cesium from SNF, although strontium appears to be insoluble. Figure 4 shows the HSC Chemistry result for the separation of strontium from SNF via dissolution in water after removal of cesium and other Group 1 ions. A second dissolution with water did not cause adequate separation of strontium from SNF; shown by SrF_2 being the largest component.

SNF after a 10-yr cooling period contains 772.5 g/MTIHM of strontium. Assuming a reprocessing plant would treat 2000 MTIHM/year with 200 operating days/year, 7950 gallons of water/hour would be required to separate the 322g Sr/hr according to HSC. Under the same conditions when the common ion effect is not considered, only 700 gallons of water would be needed based on the solubility of strontium listed in Lide (12).

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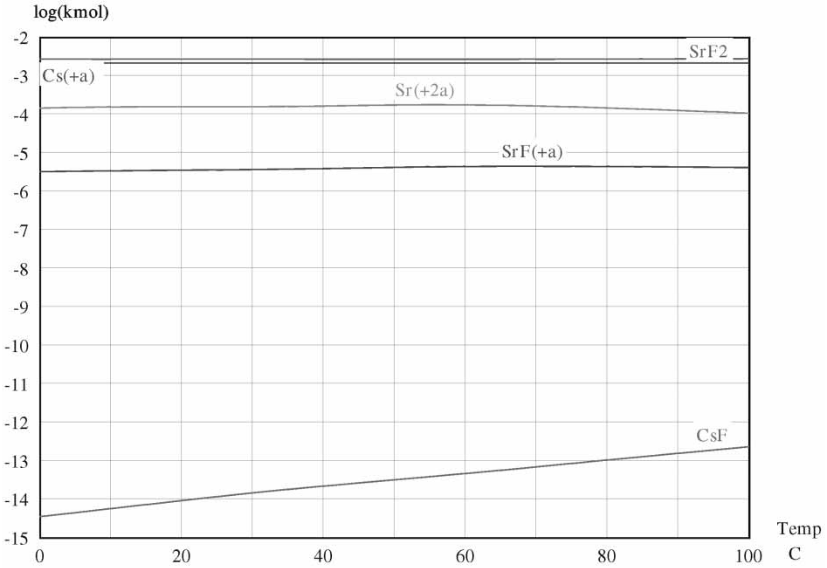

Figure 3. HSC Chemistry equilibrium composition for separation of Cs/Sr.

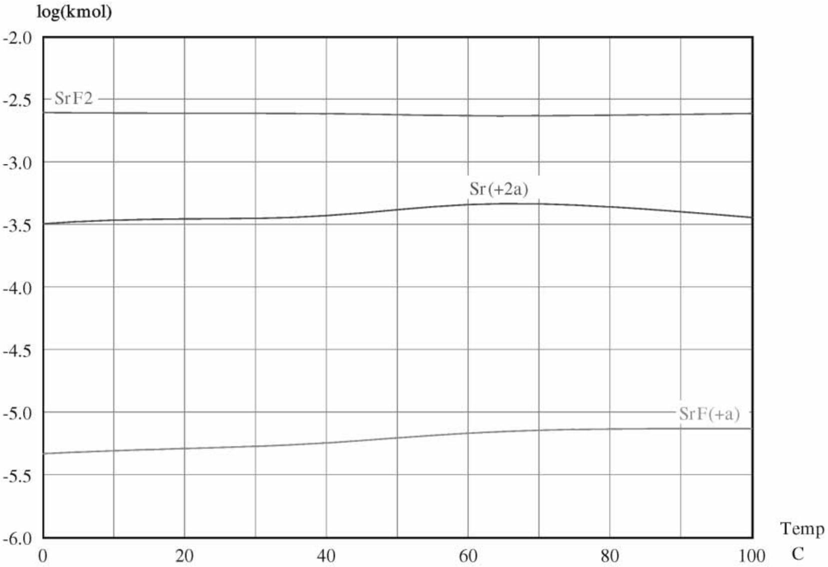

Figure 4. HSC Chemistry equilibrium composition for separation of Strontium after removal of cesium.

CONCLUSIONS

The HSC model is valid for the prediction of qualitative solubilities. Currently, HSC assumes an ideal solution. Activity coefficients are needed to improve the capabilities of HSC, or another model must be developed.

The HSC model predicts that CsF can be readily separated from SNF using water, while SrF_2 cannot be practically separated from SNF using water. Cesium and strontium can be disposed of together; hence, separation of these two nuclides is not desired. Initially, the process seemed reasonable; however, the common ion effect was predicted to prevent dissolution and separation of the strontium with the cesium. Cesium fluoride is highly soluble and does not appear to be suppressed by any ions.

Because $Sr(OH)_2$ is more soluble (0.41 g/100 mL H_2O at 0°C and 21.83 g/100 mL H_2O at 100°C) than SrF_2 (0.012 g/100 mL H_2O at 27°C), conversion to hydroxide and separation is another possibility. There are possible advantages to having strontium separate from cesium for disposal. Strontium only has one radioactive isotope, which has a half-life of 30 yrs. Cesium has one isotope with a 30-yr half-life and another with a much longer half-life. Cesium would therefore require geologic disposal, while strontium could be allowed to decay outside the repository until possible disposal as low-level waste. Therefore, more research is needed to examine the possibility of converting fluoride residues to hydroxides, after removal of the Group 1 ions, to improve the solubility of strontium. Group 1 ions are also soluble hydroxides; therefore, cesium and strontium could potentially be separated together, if desired. Further study of downstream processing after removal of strontium (for example, removal of Pu + Np) is also needed.

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