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**BUDAPEST UNIVERSITY OF TECHNOLOGY AND ECONOMICS
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Optimization of Borosilicate Glass Composition for the Stabilization of Uranium and Lanthanides

Thesis Summary

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INTRODUCTION

The use of radioactive materials is an integral part of modern life. They are applied in agriculture, the food industry, and medicine - from studying nutrient transport and food preservation to diagnostic and therapeutic procedures. These activities, as well as energy production, generate radioactive wastes whose safe management and disposal are of paramount importance.

The concepts of radiation and radioactivity are most commonly associated with nuclear reactors, which, through their safe operation, significantly contribute to electricity generation. However, during operation and decommissioning, radioactive wastes of varying activity levels are produced, among which long-lived radionuclides originating from spent fuel present the greatest challenge. The management of radioactive waste and the reduction of its volume are among the key tasks of the nuclear industry.

Continuous operation and reactor decommissioning generate increasing amounts of radioactive waste, for which several safe and long-term storage solutions exist. A socially accepted and technically justified method is disposal in deep geological repositories, where combined natural and engineered barriers ensure waste isolation from the environment. Prior to disposal, the waste must be treated and stabilized to reduce its volume and convert it into a safe, manageable form.

One of the most effective methods for stabilizing high-level radioactive waste is vitrification, in which the waste is immobilized in glass through high-temperature melting, a process referred to as waste conditioning. In France, vitrification has been an established industrial practice for decades. Experience confirms that immobilization in borosilicate glass provides a safe and reliable long-term technological solution for conditioning high-level radioactive waste.

OBJECTIVE

Among the radioactive wastes generated during nuclear power production, the storage and safe disposal of spent fuel and high-level waste from its reprocessing represent a major challenge. The first step in waste disposal is conditioning, i.e., stabilization of the waste within a matrix material. According to international consensus, the most suitable material for conditioning high-level radioactive waste is an amorphous glass matrix produced by vitrification. The use of a glass matrix is particularly advantageous for conditioning actinides (U, Np, Pu, Am, Cm), as these isotopes can be stably incorporated into the amorphous glass structure and remain chemically inert and secure over long periods.

Based on literature data, potential glass types were reviewed, and borosilicate glass was identified as one of the most effective matrix for radionuclide immobilization. These glasses possess excellent mechanical and structural properties: radionuclides are incorporated into the glass network, exhibit low leaching rates, and can be produced economically. Previous studies have shown that the $\text{SiO}_2\text{-B}_2\text{O}_3\text{-Na}_2\text{O-BaO-ZrO}_2$ oxide system can accommodate up to 30 wt% UO_3 while maintaining a stable amorphous structure without crystalline phase formation.

The aim of my research is to optimize the composition of a $\text{SiO}_2\text{-B}_2\text{O}_3\text{-Na}_2\text{O-BaO-ZrO}_2$ five-component borosilicate glass to achieve effective stabilization of various radionuclides in the highest possible concentrations. Following synthesis, the compositions are examined from structural and chemical perspectives to understand how the radionuclides incorporate into the glass network, in what proportion, and with what stability. The study focuses on actinide stabilization, using the basic composition (55) SiO_2 (25) Na_2O (10) B_2O_3 (5) BaO (5) ZrO_2 (mol%) as a reference. During the experiments, glass compositions loaded with individual actinides and lanthanides were prepared and examined step by step, followed by their combined addition to the base composition. As laboratory conditions allowed the use of uranium only, lanthanides were employed as surrogates for Pu, Am, and Cm incorporation, an approach widely accepted in the literature.

Uranium is the most important actinide component. Lanthanides (Ce, Nd, Eu) were used as chemical analogues of actinides (Pu, Am, Cm), as their similar chemical behavior allows modeling of the actinides most likely present in radioactive waste. Following a stepwise approach, the aim was to understand the incorporation of uranium into the glass matrix

structure, then that of lanthanides (as actinide surrogates), and finally the simultaneous incorporation of uranium and lanthanides into the glass network.

To investigate the structure, analytical methods traditionally applied to amorphous systems were selected, supported by extensive international literature, allowing comparison of the obtained results with previously published data. The chosen techniques - neutron diffraction, Reverse Monte Carlo simulation, XANES, EXAFS, and product consistency tests - address key aspects of structural and chemical stability. Neutron diffraction and Reverse Monte Carlo simulation reveal the short- and medium-range order of the amorphous network, while synchrotron-based XANES and EXAFS provide information on the oxidation states and local coordination environments of uranium and lanthanides. Product consistency test assess long-term chemical durability. The prepared sample series are as follows:

Lanthanide-containing glasses:

Lanthanides were used to model the chemical behavior of actinides, with Ce substituting for Pu, Nd for Am, and Eu for Cm. The glass matrices were prepared using SiO₂ and B₂O₃ as glass formers, Na₂O and BaO as modifiers, and ZrO₂ as a stabilizer. The lanthanide components used as actinide analogues were CeO₂, Nd₂O₃, and Eu₂O₃. The glass structure and lanthanide incorporation were analyzed by diffraction and spectroscopic techniques. Samples prepared by the melt-quenching technique were: (90/70) wt% [Matrix] + (10/30) wt% CeO₂, (90/70) wt% [Matrix] + (10/30) wt% Nd₂O₃, and (90/70) wt% [Matrix] + (10/30) wt% Eu₂O₃, where the Matrix corresponds to the base composition (55)SiO₂(25)Na₂O(10)B₂O₃(5)BaO(5)ZrO₂ (mol%). Lanthanide concentrations were examined between two limits to simulate vitrified waste containing lower (10 wt%) or higher (30 wt%) actinide levels. These values are consistent with previously reported international data. Neutron diffraction measurements were analyzed using Reverse Monte Carlo simulations, and the local structures of Ce, Nd, and Eu atoms were investigated by synchrotron-based techniques.

Uranium-containing glasses:

The incorporation of uranium alone into the glass matrix structure was also investigated. Sample preparation followed the same procedure as before, using UO₃ containing natural uranium to model the waste. The prepared samples were: (100-X) wt% [Matrix] + X wt% UO₃, where X = 10, 20, 30, 40 and the Matrix corresponds to (55)SiO₂(25)Na₂O(10)B₂O₃(5)BaO(5)ZrO₂ (mol%). Neutron diffraction data were analyzed

using Reverse Monte Carlo (RMC) simulations. The uranium sample series was designed to cover a wide concentration range and to determine the maximum uranium oxide content that can be stabilized within the amorphous glass matrix. The oxidation state of uranium was determined from XANES spectra recorded at the U L_{III}-edge, while its local environment was investigated using EXAFS analysis.

Lanthanide- and uranium-containing glasses:

In the next stage, the concept was extended by adding mixed lanthanide/uranium oxide blends - varying in both composition and concentration - to the base glass matrix (55)SiO₂(25)Na₂O(10)B₂O₃(5)BaO(5)ZrO₂ (mol%) to simulate waste loading and examine the mechanisms of stable incorporation using complementary structural methods. The prepared sample series were: 70 wt% [Matrix] + 20 wt% UO₃ + 10 wt% CeO₂; 70 wt% [Matrix] + 20 wt% UO₃ + 10 wt% Nd₂O₃; 70 wt% [Matrix] + 20 wt% UO₃ + 10 wt% Eu₂O₃; and 60 wt% [Matrix] + 10 wt% UO₃ + 10 wt% CeO₂ + 10 wt% Nd₂O₃ + 10 wt% Eu₂O₃. For the mixed compositions, 20 wt% uranium oxide and 10 wt% lanthanide oxide concentrations were applied, selected based on results from single-component systems (containing only uranium oxide or only lanthanide oxide) and literature data. These concentrations individually showed favorable performance and were thus suitable for studying the mutual interactions of multiple components. The equal-ratio composition (10-10-10-10 wt%) of uranium and lanthanide oxides was defined based on the favorable stability observed in the previous single-component systems with 10 wt% loading.

Since conditioned waste must be disposed of in a deep geological repository with multiple barrier systems, vitrified waste forms represent the first containment barrier. Accordingly, the long-term behavior and chemical durability of the borosilicate model waste glasses were evaluated through leaching experiments. The leaching tests were performed using Milli-Q Type 1 ultrapure water (MQ water) and a synthetic porewater formulated to match the chemical composition of the Boda Claystone Formation (BCF), considered a potential host rock for high-level waste disposal in Hungary.

Following the multi-barrier concept, a corrosion experiment was conducted to study the behavior of the glass matrix under high temperature, high pressure, and saturated conditions, focusing on surface reactivity. The vitrified waste is in contact with other materials, notably the metal container into which it is cast, as well as the backfill material - in this study, Boda

claystone. Understanding corrosion behavior helps assess the long-term performance of the waste matrix and the suitability of the selected metal container for storage. The glass matrix used had the composition (55)SiO₂(25)Na₂O(10)B₂O₃(5)BaO(5)ZrO₂ (mol%), with iron shavings simulating the metal container and powdered Boda claystone as the backfill. Surface analyses were performed using SEM/EDX and XPS, while solution analyses were carried out by ICP-OES and ion chromatography (IC).

RESULTS

Summary of results for lanthanide-containing glasses

To the borosilicate glass composition (55)SiO₂(25)Na₂O(10)B₂O₃(5)BaO(5)ZrO₂ (mol%), 10 wt% and 30 wt% of CeO₂, Nd₂O₃, or Eu₂O₃ lanthanide oxides were added separately. The glasses were characterized by neutron diffraction with RMC simulation and XANES/EXAFS analysis. Both the RMC simulations and neutron diffraction data consistently indicate that the base structure of the Ln-oxide-containing borosilicate glasses consists of mixed ³B-O-⁴Si and ⁴B-O-⁴Si units. The ratio of BO₃ to BO₄ units strongly depends on the Ln content, with BO₄ formation increasing proportionally with higher Ln-oxide concentration.

The Ce-O, Nd-O, and Eu-O bond lengths and coordination numbers, together with the partial atomic pair correlation functions between Ce, Nd, Eu, and the network-forming Si and B atoms, confirm that the lanthanide-containing glasses possess a stable structure.

The Ln XAFS spectra are in good agreement with the RMC simulation results, showing identical local structures around the lanthanides in both analyses. The partial atomic pair correlation functions $g_{\text{Nd-O}}(r)$ and $g_{\text{Eu-O}}(r)$ exhibit similar peak positions, while second-neighbor contributions derived from EXAFS notably influence the RMC values. Both experiments confirm the presence of Si and B environments around the lanthanides, indicating their incorporation into the Matrix structure.

The leaching behavior of the Matrix components and lanthanides differs, yet similar trends are observed across the sample series. The concentrations of dissolved Ce, Nd, and Eu decrease over time, indicating that the investigated compositions may be suitable for the long-term stabilization of the corresponding actinide surrogates (Pu, Am, Cm).

Summary of results for uranium-containing glasses

Borosilicate glass compositions containing uranium were investigated by adding 10, 20, 30, and 40 wt% uranium oxide to the Matrix. The glass structure was studied using neutron diffraction, RMC simulation, and X-ray absorption techniques (XANES, EXAFS) to evaluate the effect of uranium on the borosilicate glass network, with particular focus on B-O, Si-O, and Na-O environments, and to determine the mechanism of uranium incorporation. The chemical durability of the sample series was assessed through leaching experiments.

Neutron diffraction results confirm that stable amorphous compositions were obtained even at increasing uranium concentrations. RMC simulations show that the glass network is primarily composed of ${}^4\text{Si-O-}^3\text{B}$ and ${}^4\text{Si-O-}^4\text{B}$ units, with the ${}^4\text{Si-O-}^3\text{B}$ linkages becoming dominant as uranium content increases. Uranium predominantly bonds with non-bridging oxygen atoms in axial positions, which do not form active connections with other atoms such as boron or silicon. This behavior corresponds to a decrease in boron coordination number from four to three. EXAFS fitting confirmed that uranium occurs mainly as hexavalent uranyl (U^{VI}) species, with a linear $\text{O}=\text{U}=\text{O}$ configuration and four additional equatorial oxygen atoms, while U^{V} exists in octahedral coordination. The ratio of pentavalent and hexavalent uranium remained similar across the sample series.

Leaching experiments revealed significant differences in the dissolved element concentrations between samples containing 10-20 wt% and those with 30-40 wt% UO_3 . In samples with lower UO_3 content, the Si, B, and Na concentrations in the leachates were higher than in the Matrix, whereas above 30 wt% UO_3 these values were lower. The Matrix-U30 sample exhibited the lowest Si and B leaching rates among the UO_3 -containing glasses, even below those of the base Matrix. Uranium showed the lowest leaching rate in the Matrix-U30 composition across the entire series.

Consequently, it can be concluded that the investigated borosilicate Matrix is capable of accommodating up to 40 wt% UO_3 , demonstrating significant potential for high-level nuclear waste immobilization. The results confirm that even at 40 wt% UO_3 , uranium is stably incorporated into the Matrix network, and the presence of radioactive isotopes does not induce any detectable structural changes.

Summary of results for lanthanide- and uranium-containing glasses

Borosilicate glass samples containing both lanthanides and uranium were prepared, and their structures were characterized by neutron diffraction, X-ray absorption spectroscopy, and RMC modeling. In addition, PCT-B tests were performed to evaluate the chemical durability of the glass samples immersed in porewater.

Neutron diffraction and Reverse Monte Carlo simulations confirmed that the base structure of the mixed U-Ln glasses is identical to that of the Matrix, consisting of tetrahedral SiO_4 units and mixed BO_3/BO_4 groups. Similar to the U-containing samples, the combined addition of U and Ln did not cause significant structural changes relative to the Matrix. Uranium

acts as an intermediate oxide, reducing the fraction of four-coordinated boron, while Ln ions behave as network modifiers, shifting B-O coordination from threefold to fourfold. The proportion of non-bridging oxygens decreases, accompanied by the $\text{BO}_3 + \text{O}^- \rightarrow [\text{BO}_4]^-$ isomerization process.

Modeling of the U L_{III}-edge spectra considered uranium in the U^{VI} uranyl form (linear $\text{O}=\text{U}=\text{O}$ configuration with four equatorial oxygen atoms) and in U^V octahedral coordination. Compared to the previous U-containing glass samples, the U^V-O bond length increased slightly by 0.05-0.07 Å, and enhanced static disorder was observed in the U^VO₅ octahedra. The structural role of Nd was unaffected by the presence of U, Ce, or Eu. In the MUCe sample, LCF analysis confirmed 3.9% Ce^{IV} content, while the MUCNE sample showed a higher Ce^{IV} fraction of approximately 12.3%. No redox sensitivity was observed: Nd existed exclusively in the Nd^{III} state in both MUNd and MUCNE samples, and the Eu-containing glasses contained only Eu^{III} ions. LCF analysis of the U L_{III}-edge spectra did not indicate the presence of U^{IV}; the samples consistently contained U^V and U^{VI} in comparable proportions.

The PCT test results showed increased Si, B, and Na concentrations in all leachates, indicating glass dissolution. These concentrations rose with test duration for all samples, with the highest values measured in the MUNd sample. The data suggest that the MUNd composition yields a weaker glass structure, whereas the MUCNE sample exhibited the greatest structural stability during leaching.

The investigated borosilicate glass matrix is capable of stably incorporating high concentrations of combined lanthanides and uranium, making it suitable for immobilizing multicomponent radioactive waste.

Summary of results for the laboratory MGR model system

In this study, a complex model setup was constructed to examine the interactions among engineered barrier materials under simulated repository conditions over time. The experiments were conducted for 3, 7, and 12 months, followed by post-mortem analyses of the glass, iron, and clay components. The main elements of the borosilicate glass - Si, O, Na, Zr, and Ba - were all detected by EDX analysis after 3, 7, and 12 months. Although no secondary phases or alteration layers were observed on the borosilicate glass samples within the resolution of backscattered electron images at 20 keV ($\approx \mu\text{m}$ scale), slight changes in elemental composition were detected. This indicates that within the information depth ($\approx 1 \mu\text{m}$), the average

composition remained unchanged. Complementary surface analysis was performed by XPS directly on the Matrix sample to detect possible alterations or secondary phase formation. The results showed that the surface composition of Si and O (at a depth of ≈ 5 nm) remained essentially constant after 3 months, while Na and Ba concentrations decreased. Boron was not detectable within the XPS information depth. The most significant observation was the deposition of Mg and Ca from the pore solution on the borosilicate glass surface, which may contribute to the formation of a passivating layer.

According to the ICP-OES results, the amount of leached boron increases over time. Among the glass-forming elements, the dissolved Ca and Mg concentrations decrease, consistent with the XPS findings, while K, Si, and Na concentrations increase. Ion chromatography measurements showed that the concentrations of Cl^- and SO_4^{2-} ions did not increase significantly even after the extended experimental period.

The results indicate that under the tested conditions (12 months, 80 °C), the modeled components of the engineered barrier system exert minimal interaction and each retains its integrity, consistent with the principle of multiple containment. The borosilicate glass remained stable, with no formation of secondary phases. No corrosion products were identified on the iron shavings. Based on three parallel experiments, the mineral components of the BCF did not react with the other materials and slowed the structural alteration of the glass, including corrosion processes during glass-water interaction, hydrolysis of network-forming Si-O and B-O bonds, and the onset of leaching and secondary phase formation.

SUMMARY

In My dissertation, borosilicate glasses of various compositions were synthesized and structurally characterized to assess their potential applicability for stabilizing high-level radioactive waste of different types and loadings. To the base “*Matrix*” composition (55)SiO₂(25)Na₂O(10)B₂O₃(5)BaO(5)ZrO₂ (mol%), varying amounts of lanthanide oxides (CeO₂, Nd₂O₃, Eu₂O₃), uranium trioxide (UO₃), and their mixtures were added. The resulting glass compositions were subjected to structural and corrosion studies.

Structural analyses were performed using neutron diffraction, XANES, and EXAFS spectroscopy. The neutron diffraction and EXAFS data were processed by Reverse Monte Carlo simulation. The results indicate that both lanthanides and uranium are stably incorporated into the glass structure. The amorphous nature was preserved regardless of doping level, and no crystalline phases were identified. Uranium occurs in U^V and U^{VI} oxidation states, predominantly bonded to non-bridging oxygen atoms.

Among the various compositions, glasses containing only lanthanides, only uranium, or their mixtures all exhibit well-defined structures. Structural data confirm that the Matrix can stably incorporate both uranium and lanthanide atoms. No unfavorable structural rearrangement was observed in the compositions containing both uranium and lanthanides.

Chemical durability was evaluated through leaching experiments using MQ water and synthetic Boda porewater. The results showed that the network-forming Si and B atoms exhibit low leaching rates. The leaching of Ce, Nd, and Eu decreased over time, indicating their structural stabilization within the glass network. Leaching data revealed differences between samples containing 10-20 wt% and 30-40 wt% UO₃: lower UO₃-loaded samples released higher amounts of Si, B, and Na, while higher UO₃ loadings resulted in lower concentrations compared to the Matrix.

To simulate repository conditions, a laboratory-scale repository system was designed to study the interactions between glass, iron, and Boda claystone over three time intervals (3, 7, and 12 months). SEM, XPS, and solution analysis results revealed minimal interaction among the components, with the formation of an initial passivating layer on the glass surface. No detectable iron corrosion was observed.

The results indicate that the investigated Matrix possesses stable structural properties, enabling the incorporation of lanthanides and uranium at high concentrations. All samples remained resistant to chemical effects that could significantly alter the structure under the tested conditions (80 °C, porewater saturation) and time intervals. Based on these findings, the Matrix composition is recommended as a potential base formulation for conditioning high-level radioactive waste generated during reprocessing.

NEW SCIENTIFIC RESULTS**THESES**

[1]

Borosilicate glasses containing lanthanide oxides (CeO_2 , Nd_2O_3 , Eu_2O_3) were synthesized with varying Ln-oxide concentrations (10/30 wt%). Structural analysis confirmed that each lanthanide is bonded at second-neighbor distances to the network-forming Si and B atoms, indicating stable incorporation into the base structure composed of mixed $^4\text{Si-O-}^3,^4\text{B}$ units. It was established that the leaching of Si and B does not compromise the structural stability, while the concentrations of leached Ce, Nd, and Eu decreased over time, demonstrating a stable glass network.

*Related publication:*M. Fabian, F. Pinakidou, I. Tolnai, O. Czompoly, J. Osan

Lanthanide (Ce, Nd, Eu) environments and leaching behavior in borosilicate glasses

Scientific Reports 11 : 1 Paper: 13272 , 15 p. (2021)

<https://doi.org/10.1038/s41598-021-92777-w>

[2]

A borosilicate glass series containing uranium trioxide was prepared with varying UO_3 concentrations (10/20/30/40 wt%) to study uranium incorporation. Structural results confirmed that the Matrix can accommodate up to 40 wt% UO_3 - the highest concentration reported to date. Uranium is predominantly present in $\text{U}^{\text{V}}/\text{U}^{\text{VI}}$ oxidation states, and increasing UO_3 content promotes the formation of $^4\text{Si-O-}^3\text{B}$ units. Leaching experiments showed that the glass containing 30 wt% UO_3 exhibits the lowest Si, B, and U leaching rates within the series, identifying this composition as the most stable.

*Related publication:*I. Tolnai, J. Osan, F. Pinakidou, Zs. Kovacs, M. FabianEffects of UO_3 on the structure, thermal and chemical stability of borosilicate glass matrix

Journal of Non-Crystalline Solids 637 Paper: 123054 , 13 p. (2024)

<https://doi.org/10.1016/j.jnoncrysol.2024.123054>

[3]

A borosilicate glass series was prepared using a custom formulation with combined but varying additions of lanthanide oxides and uranium trioxide (20 wt% UO_3 + 10 wt% CeO_2 , 20 wt% UO_3 + 10 wt% Nd_2O_3 , 20 wt% UO_3 + 10 wt% Eu_2O_3 , and 10 wt% UO_3 + 10 wt% CeO_2 + 10 wt% Nd_2O_3 + 10 wt% Eu_2O_3). Structural analysis showed that the combined addition of U and Ln oxides did not alter the Si-O-B network structure; uranium trioxide behaves as an intermediate oxide, while lanthanides act as network modifiers. Uranium occurs as U^{VI} uranyl species (linear $\text{O}=\text{U}=\text{O}$ configuration with four equatorial oxygen atoms) and U^{V} in octahedral coordination. Lanthanides are present as Ce^{III} , Nd^{III} , and Eu^{III} . The coexisting U/Ln species do not affect each other's oxidation states. Leaching results indicated that the glass containing 10 wt% UO_3 + 10 wt% CeO_2 + 10 wt% Nd_2O_3 + 10 wt% Eu_2O_3 has the most stable structure, whereas the 20 wt% UO_3 + 10 wt% Nd_2O_3 composition shows the lowest chemical durability.

Related publication:

I. Tolnai, J. Osan, P. Jovari, F. Pinakidou, A. Sulyok, M. Fabian

Structural characterization of uranium and lanthanide loaded borosilicate glass matrix

Scientific Reports 15 : 1 Paper: 28352 , 19 p. (2025)

<https://doi.org/10.1038/s41598-025-13166-1>

[4]

A scaled engineered barrier system was designed to study the interactions among glass, iron, and clay materials in a laboratory-scale setup over 3, 7, and 12 months. Under the tested conditions (12 months, 80 °C), the borosilicate glass remained structurally stable, with Mg- and Ca-rich surface deposits observed but no secondary phase formation. No iron oxide or corrosion products were identified on the iron shavings. The clay components did not react with the glass or iron but reduced their reactivity. The results indicate that, under the examined conditions, all three components of the engineered barrier system are suitable for developing a disposal concept.

Related publication:

I. Tolnai, J. Osan, O. Czompoly, A. Sulyok, M. Fabian

Glass/steel/clay interactions in a simulated radioactive waste geological disposal system

Scientific Reports 13 : 1 Paper: 20381 , 13 p. (2023)

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