

Direct immobilisation of radioactive liquid organic waste in a geopolymer matrix

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Abstract. Managing and disposing of radioactive liquid organic waste is a complex task due to its hazardous nature and long-term environmental impact. Geopolymer materials, produced through the alkali activation of aluminosilicate precursors, have emerged as promising candidates for immobilising various types of waste, including radioactive waste, due to their chemical stability, mechanical strength, and resistance to degradation. This study investigates the feasibility and effectiveness of the direct conditioning of radioactive liquid organic waste surrogate into a blast furnace slag matrix, focusing on key properties such as mechanical strength, porosity, and durability under various curing conditions. Two experimental series were conducted, using different waste oils as surrogates for radioactive liquid organic waste and various surfactants to evaluate their impact on the geopolymers. Samples were cured under sealed and aerated conditions to assess how these variables influence the resulting material. Several methods, such as UV/Vis spectroscopy, microscopy, porosimetry and compressive strength testing, were utilised to analyse the physical, chemical, and mechanical characteristics of the produced geopolymer waste forms. Compressive strength test results indicate potential suitability for long-term storage and disposal of radioactive liquid organic waste. However, increasing waste oil and surfactant concentrations generally reduced compressive strength and were associated with increased porosity. Additionally, leaching tests were performed to evaluate the potential release of oil and selected elements from the stabilised waste form. The findings demonstrated minimal oil leaching, suggesting a high level of immobilisation efficiency. These findings indicate the potential of geopolymer matrices for encapsulating radioactive liquid organic waste. However, they also highlight the need for careful optimisation of surfactant and waste concentrations to balance the volume of incorporated waste oil and mechanical performance. Further research and optimisation are required to refine these formulations and expand the application to other liquid organic waste types, aiming to develop practical and scalable solutions for radioactive waste management.

1 Introduction

Radioactive waste generated during the operation and decommissioning of industrial nuclear facilities consists of various types of radioactive waste, including radioactive liquid organic waste (RLOW). Managing this waste, which may contain scintillation cocktails, solvents or oils [1], is challenging due to the hazardous nature and the need for long-term stability during disposal in the repository. Even though there are conventional methods of treatment, which include solidification into a cementitious matrix after absorption into a porous material or incineration [2,3], they come with disadvantages such as technological complexity and secondary waste production [4,5]. Geopolymers, as inorganic aluminosilicates, have emerged as promising matrices for the immobilisation of such wastes. These materials exhibit excellent mechanical

strength, durability [6], and resistance to acids and heat, making them suitable for immobilising radioactive waste [7]. The curing time for geopolymers is shorter than for cements because the setting mechanism of geopolymers relies on rapid polymerisation rather than hydration [8]. The versatility of geopolymers allows them to be synthesised from a variety of industrial by-products, including fly ash, metakaolin, and blast furnace slag (BFS) [9–13]. Studies have shown the applicability of geopolymers for immobilising various types of radioactive waste, including oil-based drilling fluids [4,14] or iron and sulphur-rich sludges [15,16]. Additionally, further works have shown that geopolymer matrices can incorporate up to 30% of RLOW by volume, depending on the specific formulation and the properties of the waste [7,17]. Blast furnace slag, a by-product of the iron-making process, is rich in calcium and silicate minerals, making it an effective precursor for geopolymer synthesis. The use of BFS in

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geopolymer matrices offers several advantages, including improved mechanical strength, reduced setting time, and enhanced immobilisation of radionuclides. Blast furnace slag can be activated by alkaline solutions, such as sodium hydroxide or sodium silicate, to form a stable geopolymer network [12,18]. Although the direct incorporation of RLOW into BFS-based geopolymer matrices are promising, it has not been sufficiently investigated. Therefore, in this study, we investigate the direct conditioning of Nevastane and Mogul oils, which are used as RLOW surrogates, within a BFS matrix.

2 Materials and methods

Two series of experiments with different surrogate waste oils were conducted. In the first one, a finely ground granulated BFS (Ecocem Benelux, Belgium) provided by SCK-CEN was used with locally sourced quartz sand as the added component. The alkali binder combined sodium silicate (Sigma-Aldrich) and sodium hydroxide (Penta). The waste oil used in the experiments was Mogul TB 32 S, and we evaluated the performance of three surfactants: Tween®80, sodium dodecyl sulphate (SDS) and Glucopone 600 CS UP solution (all Sigma-Aldrich). In the second set of experiments, a finely ground granulated BFS sourced from Trinec Iron and Steel Works, Czech Republic, was used with locally sourced quartz sand. The alkali binder was the same as in the first set of experiments. Nevastane EP 100 oil was used as a surrogate waste and Tween®80 as a surfactant.

The sample preparation protocol was conducted on a laboratory scale. The activating solution was prepared 24 hours before the actual preparation by dissolving 10.6 g of sodium silicate in 32.8 ml of 10 M sodium hydroxide, followed by the addition of 124 ml of demineralised water. Before the main sample preparation, a mixture of oil and a surfactant was mixed for 10 minutes using a laboratory magnetic stirring bar and a magnetic stirrer IKA®RH basic 2 at a speed of 1000 rpm. Subsequently, 325.8 g of ground BFS was mixed with the activating solution for three minutes using a dual-action laboratory mixer operating at 130–150 rpm for the spinning head and 220–250 rpm for the stirrer. The oil-surfactant emulsion was then incorporated into the BFS and activation solution mixture and mixed for an additional 10 minutes. Finally, 195.9 g of quartz sand was added and mixed for three more minutes. The overall liquid-to-solid ratio of the mixture was approximately 0.32 by weight. The waste load, expressed in wt.%, was defined as the mass of the oil relative to the total mass of the base matrix (binder, activating solution and quartz sand) before adding the waste components. Similarly, the surfactant dose was calculated as a wt.% relative to the base matrix. A thoroughly mixed geopolymer paste was cast into cubic molds, each with dimensions of 50 × 50 × 50 mm, with a single batch producing three such samples. After casting, the samples were left to harden. The samples were demolded, weighted and cured in a sealed bag environment for 28 days at laboratory temperature. The sealed bag conditions were established by placing the samples in a sealed container

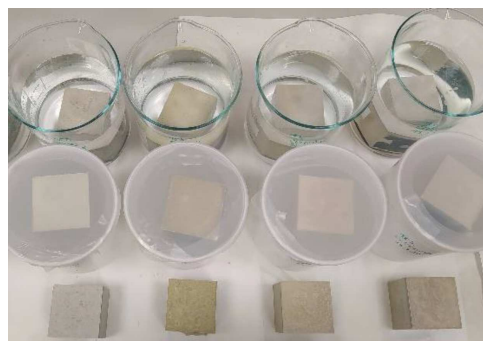


Fig. 1. Prepared BFS samples.

positioned above the surface of the demineralised water, ensuring that the sample remained untouched by the water.

After the curing period, one of the three samples of the same composition was used for leaching tests, and the remaining samples were used for compressive strength and porosity measurements. Leaching experiments were performed under static conditions by immersing each cubic sample in 400 ml of demineralised water, corresponding to a leachant-to-sample volume ratio 3.2. The leachant had an initial pH of 6.94 and conductivity of 0.094 $\mu\text{S}/\text{cm}$. Leaching was conducted at ambient laboratory temperature (approx. $22 \pm 1^\circ\text{C}$) without agitation. The leachant was replaced at set time intervals (2, 7, 14, 28, 56, and 70 days), and the collected leachates were subsequently analysed. The pH and conductivity of the leachate were measured using a WTW pH/Cond 3320 Multi-Parameter Portable Metre (Xylem Analytics). A gravimetric method was used to quantify the oil and surfactant mixture leached from the geopolymer matrix. The leachate was filtered through a dried and preweighed qualitative filter paper (KA2 type, unreinforced, 80 g/m², pore size > 8 μm). After filtration, the filter paper was dried at ambient laboratory temperature and reweighed. The mass difference between the two measurements corresponded to the amount of oil and surfactant mixture leached into the demineralised water. Calcium concentration in the leachates was determined using complexometric titration with EDTA at pH ~ 12 with murexide as an indicator, following standard procedures for Ca^{2+} quantification in aqueous samples. Silicon and iron concentrations were measured using a Jenway 6850 UV/Vis Spectrophotometer (Cole-Parmer Instrument Company). Silicon was analysed following ČSN EN ISO 16264: water quality – determination of soluble silicates by flow analysis and photometric detection, while iron concentrations were determined using the spectrometric method based on 1,10-phenanthroline, following ČSN ISO 6332: water quality – determination of iron – spectrometric method using 1,10-phenanthroline.

The compressive strength of the cured samples was tested using an MTS 300 Exceed® device according to the Czech National Standard ČSN EN 12390-3 Testing hardened concrete – Part 3: compressive strength of test specimens. The porosity of selected samples containing Nevastane oil was measured using an AutoPore IV 9500

mercury intrusion porosimeter (Micromeritics). Before analysis, the samples were dried at 35°C in the laboratory oven dryer until a constant weight was reached. Two to three fragments were selected per sample from partially crushed specimens, each weighing between 1.0 and 1.2 g. During pre-analysis preparation, the samples were evacuated until a pressure of 50 mmHg was achieved, after which the evacuation continued for an additional 30 minutes to ensure pressure stabilisation. Mercury intrusion was then performed in the pressure range of 0.005–400 MPa. The samples were also analysed for the specific surface area using the 3Flex Adsorption Analyser (Micromeritics). Before the analysis, the samples were degassed under vacuum at 30°C for 48 hours using a VacPrepTM061 (Micromeritics) system to ensure clean surface conditions for gas adsorption measurements.

Before XRD analysis, the samples were manually crushed and ground into a fine powder using an agate mortar and pestle, then back loaded into the standard sample holder for measurement. Diffraction patterns of raw BFS, a standard sample and a sample with oil were collected with a Malvern PANalytical Empyrean series 3 diffractometer, equipped with a conventional X-ray tube (Co K α radiation, 40 kV, 30 mA, line focus), multicore optics and a linear position sensitive detector PIXCel3D detector. In this case, a conventional Bragg Brentano geometry was used with the iCore optical module set with a 0.03 rad Soller slit, a 0.25° divergence slit, and a 14 mm mask in the incident beam. The dCore optical module set with a 0.25° anti scatter slit and 0.04 rad Soller slit was used in the diffracted beam. X-ray patterns were collected in the range of 5–85 deg 2 θ with the step of 0.013 deg and 600 s/step, producing a scan of about 4 hours 11 minutes. XRD patterns were not pre-treated before interpretation, as no background correction was needed. Qualitative analysis was performed with the HighScorePlus software package (Malvern PANalytical, The Netherlands, version 5.2.0) [19] and the PDF-4+ database [20]. All the samples were also analysed using a stereoscopic microscope model Motic SMZ 171 T-LED equipped with a MOTICAM S12 camera (Fig. 1).

3 Results and discussion

In the initial experimental phase, we used a waste load of 5 wt.% while investigating the performance of three different surfactants – SDS, Glucopone, and Tween®80 – at varying concentrations of 0.15, 0.3, 0.45 and 0.75 wt.%. The surfactant concentrations 0.15 and 0.3 wt.% produced unsatisfactory results, and although the samples demonstrated sufficient hardening, a significant oil release occurred. In response, we increased the surfactant levels to 0.45 and 0.75 wt.%, increasing homogeneity and decreasing oil release from the sample during the hardening period. During these experiments, challenges arose while using Glucopone and SDS. As the performance of these two surfactants was not satisfactory, we proceeded with Tween®80 only. During the initial experiments, we also observed that we had to increase the percentage of surfactant with increasing waste load. After the initial

phase, we prepared samples with the gradual increase of Mogul in one set of samples and Nevastane in the second set, as shown in Table 1. All samples showed good homogeneity with negligible to no oil release after casting. From each composition, we prepared three samples; all were cured in a sealed bag environment for 28 days. To evaluate the efficiency of waste incorporation, we calculated the volumetric loading factor, which is defined as the ratio of the waste volume (oil and surfactant) to the volume of the final solidified form.

XRD analysis

The XRD patterns of both types of BFS are shown in Figure 2. The BFS obtained from Ecocem Benelux, Belgium, primarily contained amorphous SiO₂ with traces of CaCO₃. In the BFS supplied by Trinec Iron and Steel Works, the Czech Republic also contained amorphous SiO₂, traces of CaCO₃, and minerals containing calcium, magnesium, and silicon.

Subsequently, a cured standard sample, prepared without any added oil or surfactant and a sample with Mogul oil 5 wt.% were analysed (Fig. 3).

Minerals like quartz, feldspar, and mica can be associated with incorporated sand in geopolymer samples. Additionally, tobermorite and hydrotalcite are commonly associated with the reaction products of the geopolymerisation process, forming as a result of alkali activation of the BFS. The presence of these phases indicates the development of a stable geopolymer matrix, consistent with findings in other alkali-activated systems [21] and supported by thermodynamic modelling of slag-rich formulations [22]. Identifying wollastonite in the sample suggests a localised high-temperature event during synthesis or curing. Although it should be noted that wollastonite typically forms at elevated temperatures, which are usually not achieved during the geopolymerisation process [23], its presence in this context is consistent with findings that reported wollastonite as a product in alkali-activated BFS systems subjected to extended steam curing conditions at moderate temperatures [24].

Stereoscopic microscopy analysis

A stereoscopic microscopy analysis was performed on all samples. Figure 4 shows two samples with BFS from Ecocem Benelux, and Figure 5 shows samples with BFS from Trinec Iron and Steel Works. A magnified view of the grains of quartz sand incorporated into the samples was visible in all images. We detected the presence of fractures within the samples, particularly concentrated towards their edges. A contrast in colour was evident between the surface and near-surface regions of the samples, probably due to the influence of air during drying before the analysis. The standard sample displayed significantly smaller and less frequent pores than those with added waste oil, while samples with added waste oil exhibited larger and more pronounced pores.

Nitrogen physisorption and porosity analysis

The nitrogen physisorption analysis was conducted to evaluate the pore structure of the prepared samples. The standard sample had the highest BET specific surface area, indicating a standard geopolymer mesoporous

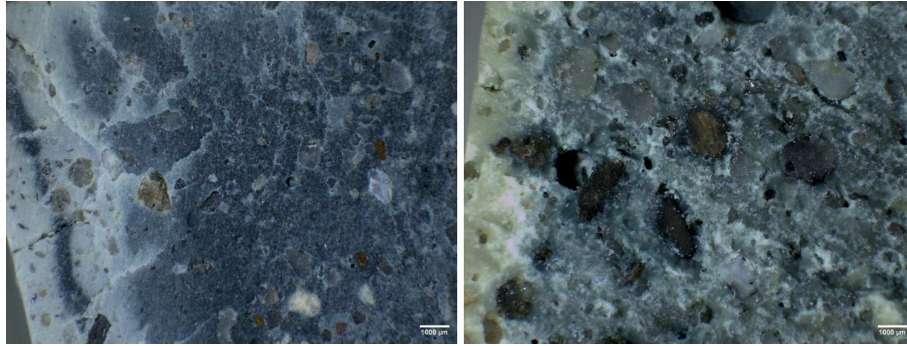


Fig. 4. Stereomicroscope images of samples with BFS from Ecocem Benelux, with no oil (left) and the sample with Mogul and surfactant mixture (right).

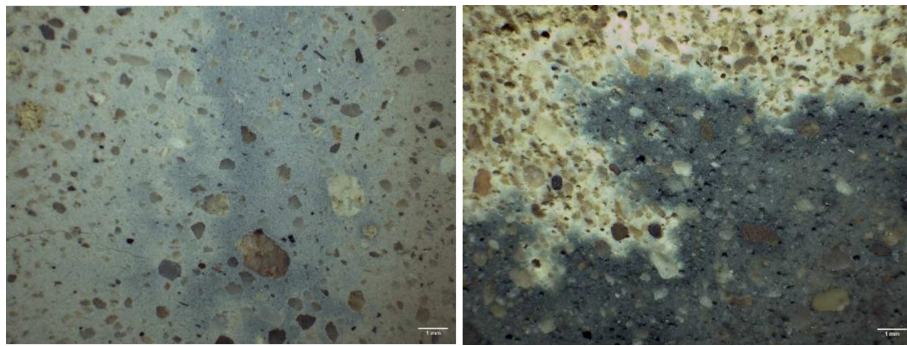


Fig. 5. Stereomicroscope images of samples with BFS from Trinec Iron and Steel Works, the sample with no oil (left) and the sample with Nevastane and surfactant mixture (right).

Table 2. Pore properties of the samples with Nevastane.

Sample	Specific surface area BET (m^2/g)	Cumulative pore volume D-H (cm^3/g)	Average pore diameter (nm)*
Standard	2.8317	0.018331	11.8380
N1 (5 wt.%)	0.1008	0.000018	1.9349
N2 (10 wt.%)	0.5274	0.000774	5.3077
N3 (20 wt.%)	0.1696	0.00006	2.5689
N4 (30 wt.%)	0.1429	0.000019	1.9984

*From D-H desorption, 4V/A, 1.7–300 nm.

carbon measurement (TOC) challenging. Although limited TOC measurements were performed, the results were inconsistent, likely due to sample heterogeneity and the limitations of our TOC analyser, which is optimised for clean aqueous samples. Therefore, we opted not to rely on these measurements. Though less commonly reported in the literature, the filtration-based gravimetric method was adopted based on internal protocols shared with other laboratories working with similar liquids. Moreover, according to safety data sheets [30,31], both Nevastane and Mogul are water-insoluble and immiscible with water, supporting the suitability of a filtration-based separation and quantification technique for their detection in leachates.

The demineralised water, where the samples were immersed, was replaced and analysed at intervals of 2, 7, 14, 28, 56, and 70 days. The results indicated that most oil was leached during the initial 14 days, after which the rate

of oil release significantly decreased. This trend was particularly noticeable in samples with a higher waste load, where the leaching rate and the total amount of leached oil were more pronounced. The cumulative oil leaching over the tested period remained below 3% for all analysed samples, indicating effective immobilisation of oil within the aluminosilicate matrix. These results are within the waste acceptance criteria (WAC) set by the national regulatory authority of the Czech Republic. For products with a volume activity above $2.107 \text{ Bq}/\text{m}^3$, no more than 4% of the total gamma-emitting radionuclides represented by the sum of ^{60}Co and ^{137}Cs may pass into the leachate. No specific leaching limit is set for products with lower activity [32]. The leaching trend is illustrated in Figure 7 and corresponds with the findings that geopolymers have been shown to exhibit low leachability, indicating their potential for immobilising radioactive waste [5,7]. While

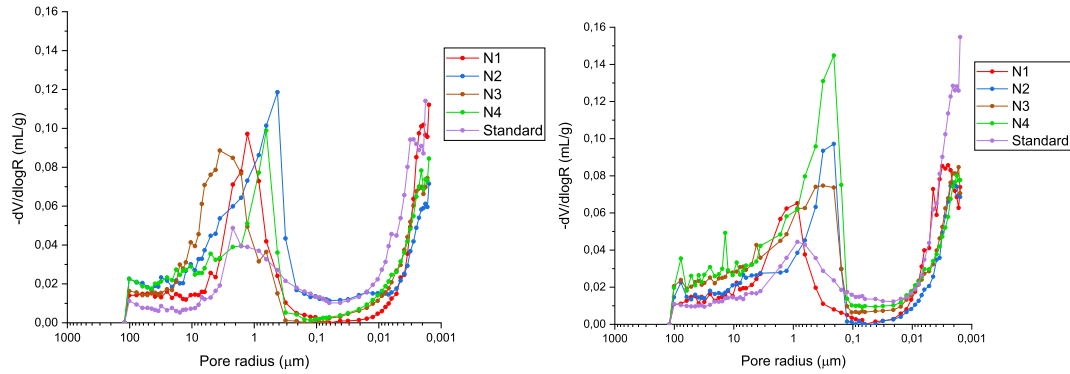


Fig. 6. The porosity of the sample without oil and samples with Nevastane before leaching (left) and after leaching (right).

all leaching data points include an individual uncertainty assessed from replicate measurements, error bars are not displayed to maintain figure clarity.

Following the analysis of oil leaching, we assessed the leaching of calcium and silica, as these elements are fundamental parts of the geopolymer structure, and tracking their leaching provides insights into potential impacts on structural integrity and mechanical properties over time. Although iron leaching was also included in the analytical scope, the measured concentrations in the leachate remained consistently below the limit of the quantification method (0.05 mg/l) and are therefore not reported further.

Calcium leaching exhibited a consistent upward trend throughout the experiment across all samples. However, the increasing rate was less steep in samples with Nevastane oil, and during the latter half of the leaching period, there was a noticeable slowing down in the leaching process. The impact of the waste load on calcium leaching was only minor in Nevastane samples (N1–N4), while higher levels of waste load correlated with a slight decrease in calcium leaching rate in samples with Mogul (M1–M3). The leached silica concentrations were higher in the samples with Nevastane. However, there was no apparent influence of waste load. The leaching slowed down after the initial increased release of silica between days 7 and 14. The cumulative leaching of calcium and silica is shown in Figure 8.

The electrical conductivity and pH were measured in all leachates (see Fig. 9). Conductivity values were consistently higher in leachates from the samples with Mogul (M1–M3) compared to those from Nevastane samples (N1–N4). There was no significant difference in conductivity across samples with different waste loads. This trend can be attributed to the differences in BFS composition. The BFS used in the M series contained higher levels of K, which could contribute to ionic strength in solution, particularly in the early stages of leaching. This is supported by the rapid decrease in conductivity observed between days 2 and 14, likely reflecting the early release of soluble ions from weakly bound phases or unreacted alkali components [33,34]. After this phase, the conductivity values stabilised. As expected, the pH values of leachate remained in the alkaline range and differed only slightly between samples. However, a gradual

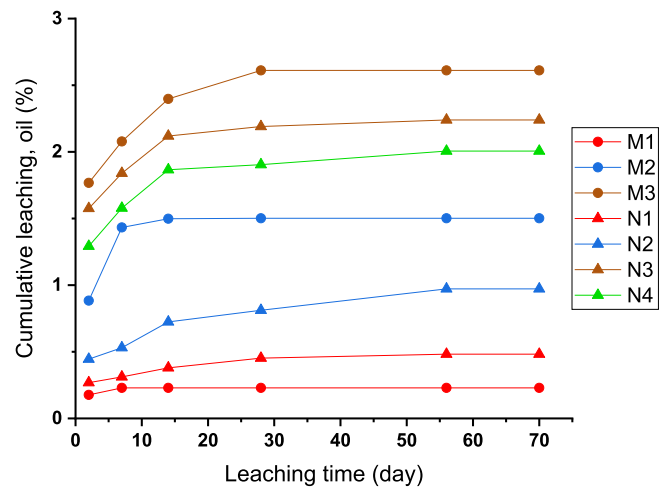


Fig. 7. Cumulative oil leaching from samples with Mogul (M1 5 wt.%, M2 10 wt.%, M3 20 wt.%) and Nevastane (N1 5 wt.%, N2 10 wt.%, N3 20 wt.%, N4 30 wt.%). The uncertainties range from ± 0.02 to ± 0.82 % across all samples and time points.

decrease in pH was observed toward the end of the leaching period.

Compressive strength

The compressive strength of the geopolymer waste form is a critical parameter for ensuring the integrity of the matrix during transportation, storage, and final disposal, and is one of the parameters of WAC. Radioactive waste solidified with an aluminosilicate matrix must achieve a minimum compressive strength of 10 MPa if its activity exceeds 2.107 Bq/m^3 [32]. Studies have shown that BFS geopolymers have high compressive strength, typically ranging from 10 MPa to over 60 MPa, depending on the formulation and curing conditions [7,12,18]. After the leaching period concluded, the samples were dried at laboratory temperature, followed by weighing and measuring compressive strength. Table 3 presents a comparison of compressive strength values before and after leaching. Introducing even a small quantity of oil into the geopolymer matrix reduced compressive strength, with further increases in oil content leading to progressively

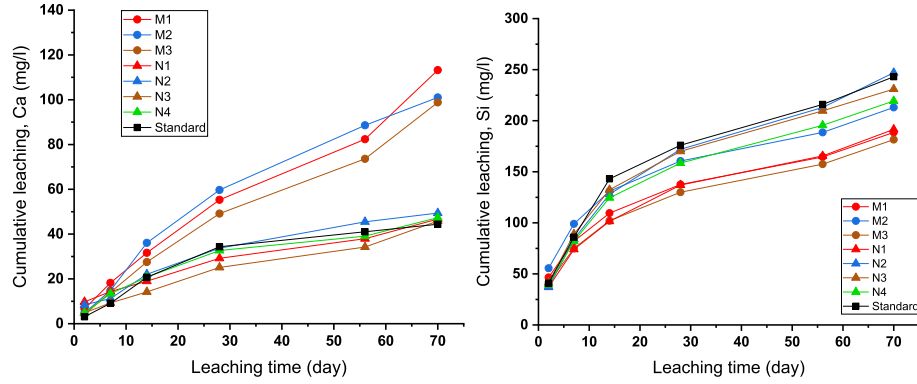


Fig. 8. Cumulative calcium (left) and silica (right) leaching from samples with Mogul (M1 5 wt.%, M2 10 wt.%, M3 20 wt.%) and Nevastane (N1 5 wt.%, N2 10 wt.%, N3 20 wt.%, N4 30 wt.%). Uncertainties for cumulative calcium leaching range from ± 0.01 to ± 13.66 mg/l and for silica from ± 2.12 to ± 53.3 mg/l, across all samples and time points.

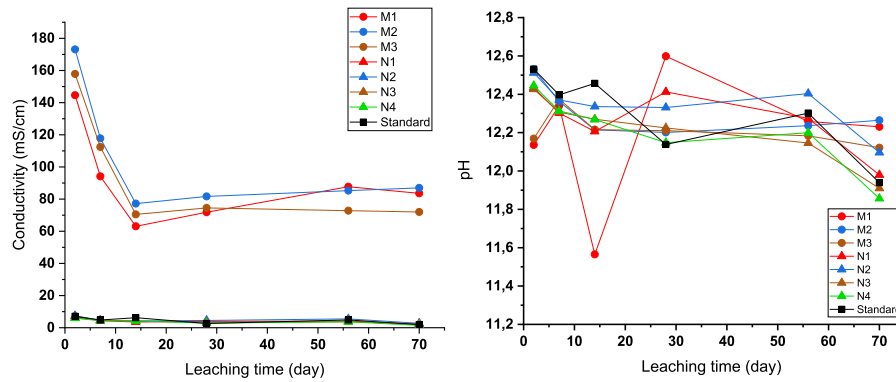


Fig. 9. Electrical conductivity (left) and pH values (right) of samples with Mogul (M1 5 wt.%, M2 10 wt.%, M3 20 wt.%) and Nevastane (N1 5 wt.%, N2 10 wt.%, N3 20 wt.%, N4 30 wt.%) during leaching. Uncertainties for conductivity measurements range from ± 0.21 to ± 8.06 mS/cm, and for pH measurements from ± 0.003 to ± 0.22 , across all samples and time points.

Table 3. The comparison of compressive strength in samples before and after leaching.

Sample type	Compressive strength (MPa) before leaching	Compressive strength (MPa) after leaching
Standard	34.30	30.56
M1 (5 wt.%)	20.71 ± 0.2	24.96 ± 1.63
M2 (10 wt.%)	18.73 ± 0.88	17.48 ± 1.68
M3 (20 wt.%)	12.14 ± 0.25	12.50 ± 0.45
N1 (5 wt.%)	19.29 ± 1.35	23.43 ± 0.55
N2 (10 wt.%)	8.63	10.20
N3 (20 wt.%)	2.15 ± 0.35	2.83 ± 0.21
N4 (30 wt.%)	1.27 ± 0.22	1.25 ± 0.15

lower values. This decrease depended on the waste loading and the type of oil used. Nevastane samples exhibited significantly lower compressive strength at higher concentrations than those prepared with Mogul. This difference can be partially attributed to the impact of oil viscosity on the geopolymer matrix. Higher viscosity oils, such as Nevastane (kinematic viscosity ~ 100 mm²/s at 40°C), can negatively impact homogeneous dispersion, which could lead to weakening of the hardened matrix. In contrast, Mogul, with a lower viscosity range of 28.5–35.2 mm²/s,

is less likely to disrupt the matrix formation to the same extent. Similar effects have been reported in geotechnical studies, where oils with higher viscosities led to greater reductions in mechanical parameters of contaminated soils [35]. However, the curing duration influences the compressive strength values and extending the curing time may increase compressive strength [36,37]. Compressive strength was determined for most formulations as the average of duplicate samples and is reported with standard deviation. In cases where only a single undamaged sample

was available (standard and N2), the results are reported as a single value without statistical dispersion. These data points are retained to represent all tested compositions in the final analysis.

The 70-day leaching period affected the compressive strength of the samples in various ways. For the Mogul samples, M1 exhibited an increase in strength post-leaching, which could suggest a potential structural rearrangement or selective leaching of weaker components. However, the other two samples displayed minimal changes. In contrast, three out of four samples with incorporated Nevastane showed increased compressive strength after leaching. Notably, only the N1 sample with a 5 wt.% waste load was within the WAC value limit. Despite the observed variability in compressive strength values before and after leaching, all samples containing Mogul met the WAC for the disposal of radioactive waste in terms of compressive strength characteristics. Additional research is needed to increase the overall compressive strength of these samples, as the hydration of BFS can be further improved by the presence of secondary activators, such as calcium hydroxide or calcium sulphate. The incorporation of these activators has been shown to enhance the mechanical properties of similar matrices [18].

4 Conclusion

- BFS-based geopolymers demonstrated potential for direct immobilisation of RLOW.
- Introducing even a low oil concentration into the BFS matrix reduces compressive strength and significantly decreases surface area and pore volume. Therefore, the balance between waste load and acceptable properties must be found.
- Surfactants minimise oil release during the curing process, but the concentration and type of surfactant must be optimised.
- Leaching tests indicate minimal oil release over time.
- Despite some variability in compressive strength, the results show that the samples containing Mogul meet WAC for radioactive waste disposal. However, further research is needed to improve the mechanical properties and scalability of the formulations.

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Conflicts of interest

The authors have nothing to disclose.

Data availability statement

The authors confirm that the data supporting the findings of this study are available within the article.

Author contribution statement

Conceptualisation, Anna Sears; Methodology, Anna Sears, Vojtěch Galek; Software, Anna Sears, Vojtěch Galek; Validation, Anna Sears, Vojtěch Galek; Formal Analysis, Anna Sears, Vojtěch Galek; Investigation, Anna Sears, Vojtěch Galek, Petr Pražák and Martin Vacek; Data Curation, Anna Sears, Vojtěch Galek; Writing – Review & Editing, Anna Sears, Vojtěch Galek; Visualisation, Anna Sears, Vojtěch Galek.

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