# An efficient and environmentally friendly

# geopolymer package for encapsulation of nuclear

# grade resins loaded with 134Cs radionuclide

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**Abstract**

Safe disposal of nuclear wastes is of great importance with the rapid development of nuclear industries. Although cementation has become the standard radioactive waste treatment strategy, Portland cement-based matrices solidification of nuclear-grade organic resins results in adverse reactions may arise with ion-exchange resin and an increase in the volume of the packaged waste. The paper explores an efficient and environmentally friendly approach for encapsulating nuclear-grade IERs using a geopolymer package, which was designed via one-factor-at-a-time concept use different un-calcined phosphate sludge (UPS) content in Metakaolin (MK) reagent. Various amounts of non-radioactive resin beads loading ranging from 9, 11, 13 to 15 wt.% within the geopolymer packages were performed. Chemical stability, mechanical properties and leaching rate of Cs+ from the individual MK and MK-UPS binary geopolymer package solidifying radioactive resins were investigated and compared to the conventional PC binder. The results indicate that the loading ratio of spent resin beads up to 13 wt.% by geopolymer package was practicable. The compressive strength of the elaborated package was greater than the waste acceptance criteria (31.55 MPa). Leaching results showed that, the leaching rate of Cs+ across 14 days reaches 2.66×10-8 cm/s for PC packages, and 1.58×10-09 cm/s for UPS-based geopolymer packages, respectively. These values were lower when compared with previously convectional PC binders, indicating the excellent solidification performance of the UPS-based geopolymer packages for radioactive resins doping with cesium. The study provides a promising paradigm for the effective solidification of radioactive grade resins and upcycling of phosphate tailings.

## INTRODUCTION

The management of large volumes of radioactive wastes, generated primarily by industrial sector and urban activities, is considered to be one of the most issues today from a global environmental and economic perspective [1]. Radioactive ion-exchange resins (IERS) are one of the simulated Intermediate Level Waste (ILW) loaded by large amounts of radionuclides including cesium (134Cs and 137Cs; half-life of 2.06 and 30.17 years, respectively) and strontium (90Sr; half-life of 29 years) [2, 3]. The radioactive resin beads are a kind of macromolecular copolymer solid materials, commonly in the form of strongly acidic (cationic) and/or weak basic (anionic) spherical beads with various particle size distributions [2]. These beads are extensively the most common techniques used for the purification of water circuits in nuclear power plants, but their quantities have been increased year after year and therefore should be properly managed using suitable ways. The Kingdom of Morocco holds 2 MW MARK-II TRIGA research reactor at Maâmora Nuclear Research Center (CENM), where annual resin beads have been estimated at 0.1 m3/years in the purification pool water of the reactor [4]. In this scope, international Atomic Energy Agency (IAEA) recommends the direct solidification of the spent IERs through cementation technology like using Portland Cement Binder (PCB), to meet the waste criteria for disposal [5]. Throughout the literature, it is true that cement-based matrices are already available and at a lower cost. However, most common PCB-based materials are highly porous, swelling and relatively easily cracked under aggressive environment with poor mechanical and thermal stability [3]. Consequently, the retention of simulated radioactive nuclides does not work well with cement matrix.

Few recent studies have found that geopolymer binders may be a better matrix than PCB for the immobilization of radioactive wastes [3, 6, 7]. For instance, Naggar et al. assess the performance of individual, binary and ternary blends of metakaolin, feldspar and blast furnace slag -based alkali activated binders to immobilize of a nuclear grade cationic resin loaded with 134Cs, 60Co and 152+154Eu radionuclides [8]. Relatedly, Lin et al. and Lee and co-workers solidified radioactive resins from Taiwan Research Reactor using MK/ blast furnace slag-based geopolymer obtaining material composites of good mechanical properties and limited release of Cs+ and Sr2+ [6, 7]. The superiority of geopolymer binder immobilization is primarily attributed to its main reaction products- as N-A-S-H, (C,N)-A-S-H, C-S-H and C-A-S-H. These reaction products are of particular interest to radioactive waste solidification owing to their ability to incorporate radionuclide ions and produce fibrous crystal interlocking through their unique amorphous network to semi-crystalline structures [9]. However, the shortage of high-quality raw materials for manufacturing geopolymer binders, as well as its high production costs, have significantly limited its production and application especially in nuclear waste treatment.

At the other end of the spectrum, by means of Moroccan phosphorus enrichment, phosphate concentrate production gave rise to 28.1 million metric tons phosphate mine tailings in the process of mineral processing in 2010 [10]. These generated tailings are accumulated in large quantities or discharged into rivers and farmlands and pose a growing threat to environmental security due to lack of effective governance, indicating that innovative phosphate mine tailing upcycling technology is urgently needed. Earlier work from our laboratory showed that the use of phosphate mine tailings can potentially produce a high performance geopolymer binders [11, 12]. However, none of the previous studies have explored the combination of phosphate mine tailings-derived geopolymer package and radioactive resins disposal.

Presented herein is an innovative and technical approach for solidifying radioactive resins using phosphate mine tailings-derived geopolymer packages. The geopolymer packages were first prepared by one-factor-at-a-time method use uncalcined phosphate sludge, which was then employed to directly solidify radioactive resins loaded with a simulated Cs. The performance assessment criteria were based on a combination of mechanical properties (compressive strength) alongside leaching rate of Cs radionuclide of the solidified packages. Collectively, the work showed the coordinated assimilation of phosphate mine tailings and nuclear wastes, which supports current thinking toward to green chemistry and circular economy.

## EXPERIMENTAL PROCEDURES

### *Materials*

The geopolymer packages are manufactured from a mixture of commercial soda silicate, adjusted with NaOH and KOH, diluted with demineralised (DI) water and eventually added with a variable proportion of un-calcined phosphate tailings (UPT) and metakaolin (MK).

The liquid sodium silicate used was used with a composition given by the manufacturer: 55 wt.% water, 29.69 wt.% SiO2 and 15.3 wt.% Na2O. The sodium and potassium hydroxides used are in the form of micro-beads, and are 99%, 90% pure, respectively. The chemical reagents were all of technical grade and were supplied from Cadilhac Company.

Phosphate tailing by-products were sampled from the Moroccan phosphate mine industry (Gantour basin, Morocco). These tailings are derived from the beneficiation of phosphate rock (washing and flotation techniques). Multiple samples were taken from a large stockpile and mechanically crushed to obtain phosphate tailing powder with a size of less than 0.08 mm, the particle size distribution was measured by a laser particle size analyser (Mastersizer 2000); the obtained powder was used in this investigation without thermal activation.

The metakaolin (MK) precursor powder was prepared from kaolin (Sibelco Company, UK) by calcination at 750°C in an electric muffle furnace (Nabertherme). The compositions and characteristics of the powders and alkaline solution are given in **Table 1**. The chemical composition of the started powders was performed using Bruker S1 Turbo SD, X-ray fluorescence (Portable System) sequential spectrometer.

TABLE. 1 Chemical composition and characteristics of reagents

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
|  | Sio2 | Al2O3 | CaO | Fe2O3 | P2O5 | MgO | Na2O | K2O | H2O | d50  (mm) |
| UPT | 10.44 | 2.38 | 42.63 | 1.32 | 11.86 | 3.10 | 0.03 | 0.25 | 0 | 0.03 |
| MK | 52.60 | 39.07 | 0.09 | 0.94 | 0.05 | 0.34 | - | 2.79 | 0 | 0.006 |
| Soda silicate | 29.69 | - | - | - | - | - | 15.3 | - | 55 | - |
| NaOH | <1 | <1 | <1 | <1 | <1 | <1 | 99 | <1 | <1 | - |
| KOH | <1 | <1 | <1 | <1 | <1 | <1 | <1 | 90 | <1 | - |

### Preparation of simulated resin beads

The ion-exchange resin (IERs) used in this study were a product of Purolite Co. (NRW-3240) supplied by German company RESINTECH INC. The examined nuclear grade resins were employed in their unloaded H+ form cationic resin and OH- form anionic resin, having a capacity indicated by the provider of 1.8 and 1 eq/L, respectively. The IER wastes are collected from the TRIGA MARK II research reactor located at Maâmora Nuclear Research Center (CENM), during the purification of the water circuits and spent fuel storage pools. They are stored with very high moisture in drums in the storage department. To determine the moisture content, the resin beads are weighted and dried in an oven at 105°C until a constant weight was reached (mf) (in triplicate) [13]. The moisture content is expressed by the following formula: Moisture (wt.%) = (mi-mf/mi) \*100.

To complete the study, leaching experiments of 134Cs incorporated into the resin beads before its solidification were undertaken. The activity of cesium was prepared with 257.98 Bq/Kg in aqueous solutions, which was obtained by dissolving CSCl in deionized water. IER wastes content were mixed and equilibrated individually -with 10mL of the aqueous solution of 134 Cs. After few days, a gamma spectroscopy (Canberra hyper-pure germanium detector (HpGe), efficiency of 30%) was utilized to determine the activities of [cesium](https://www.sciencedirect.com/topics/earth-and-planetary-sciences/caesium) at the end of the ion exchange sorption process. The artificial radionuclide, 134Cs, in the form of cesium chloride (CsCl) (analytical reagent grade 99%), was obtained from commercial suppliers Maâmora Nuclear Research Center (CENM).

### *Solidification procedure*

Geopolymer package elaboration was performed using an alkali dosage of 4.5 wt% Na2O+K2O. The soluble silicates in the activator solution were adjusted to achieve a SiO2/M2O activators modulus (Ms) of 1.3. Such alkaline solution was applied to produce the packages at different liquid/solid of about 0.90 to 1, depending on the raw mixtures. Individual and binary binders were designed according to **Table 2** by the blending of different ratios between MK to UPS. These synthesis parameters are chosen based on previous studies [11, 12], which show reliable workability and mechanical performance. Different contents of the non-radioactive resin beads (NR) (9, 11,13 and 15 wt.%) were added according to the design given in **Table 2**. All formulations were casted into cylindrical plastic moulds and were subjected to 28-day at 55°C.

The binders were designed using the one-factor-at-a-time concept to achieve the main objective of obtaining geopolymer packages with better mechanical performance and lower fractional leached activities for solidification of spent IERs. Thus, the formulationswere divided into two main groups, one of which (25Ø×52 mm) was allocated to the non-radioactive resin (NR) beads compressive strength investigations; while, the second (42Ø×86 mm), noted here radioactive resins (RR) beads, was allocated to investigate the leachability of 134Cs radionuclide matrices. The detailed package ID and proportions are given in **Table 2**, with the following nomenclature: MK/(content of UPS in wt.%) - (wt.% of non-radioactive resins used) NR/ RR .

TABLE. 2 Chemical composition of IERs-contained geopolymer packages (wt.%)

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Synthesis parameters | Packages ID | Design parameters | |  | Materials | | | | |
| MS modulus | M2O content | MK | UPS | | IERs | **134Cs** activity, Bq/Kg |
| Control package | MK/42P-0NR | 1.3 | 4.5 | 58 | | | 42 | 0 | 0 |
| Use UPS | MK/0PS-13NR | 1.3 | 4.5 |  | 100 | 0 | | 13 | 0 |
| MK/30PS-13NR | 1.3 | 4.5 |  | 70 | 30 | | 13 | 0 |
| MK/50PS-13NR | 1.3 | 4.5 |  | 50 | 50 | | 13 | 0 |
| Use of IER wastes | MK/42PS-9NR | 1.3 | 4.5 |  | 58 | 42 | | 9 | 0 |
| MK/42PS-11NR | 1.3 | 4.5 |  | 58 | 42 | | 11 | 0 |
| MK/42PS-15NR | 1.3 | 4.5 |  | 58 | 42 | | 15 | 0 |
| Use 134Cs | MK/0PS-13RR | 1.3 | 4.5 |  | 100 | 0 | | 13 | 257.98 |
|  | MK/42PS-13RR | 1.3 | 4.5 |  | 58 | 42 | | 13 | 257.98 |

MK=Metakaolin; UPS=Un-calcined phosphate sludge; NR=Non-radioactive Resins; RR= Radioactive Resins.

### Characteristics of geopolymer packages

#### Chemical stability

After curing for 28 days, IERs contained geopolymer package were immersed in deionized water (DI) with 1.5 µS/cm electric conductivity in sealed polyethylene bottles in an indoor ambient environment for 60 days. The volume of de-ionized (DI) water was 10 times the surface area of the package. After immersion, the packages were subjected to compressive strength tests.

#### Compressive strength test

After 7-28 days curing, the package compressive strength measurements were recorded using a compressive machine (brand press Carver brand model (4350.L), S/N (4350-362).

#### Leaching tests

If the elaborated geopolymer of IERs is to be accepted as a stable and suitable package for eventual disposal it is essential to show that it prevents the flow of radionuclide elements into the biosphere. The most frequently used leaching method was the Toxicity Characteristic Leaching Procedure (TCLP), as a standard toxic leaching method, which is used to test dissolution and migratory aptitude of radioactive wastes in waste packages [14], and it can be used for simulation of extreme environment solutions.

To demonstrate this purpose, leaching of 134Cs, from the examined geopolymer packages into water solution, was performed by the IAEA standard static leaching test [15]. The cylindrical monolith sample (42Ø×86 mm) was placed into the centre of a leaching vessel and mixed with DI water to maintain a solid-to-solution ratio of 10 ±1 mL of eluant per cm2 of sample. To track the 134 CS leaching rate (Ri), the leachants were replaced and analysed for radioactivity after 1d, 3d, 7d, 14d, 21d, 30d and 50d of immersion at ambient temperature. Leachates were analysed by gamma-ray spectrometry to calculate the leaching rate (Ri) which was calculated according to the following **Eq. (1)** [15–17]:

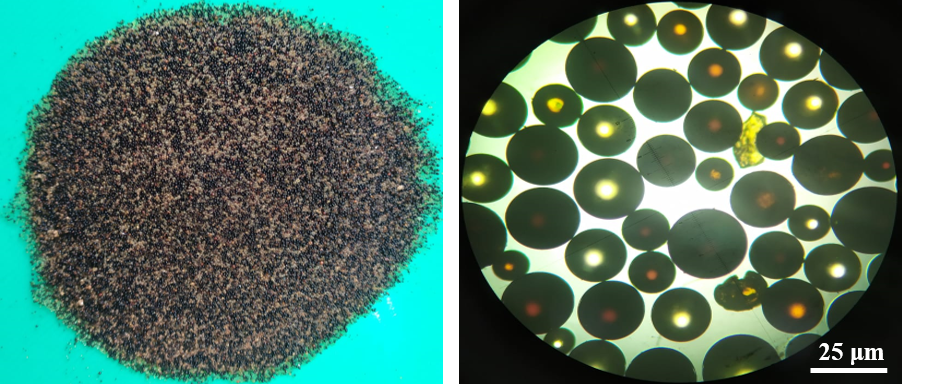
(1)

where Ri is the incremental leaching rate (cm/s), Ades is the 134Cs cumulative activities leached during each leaching interval (Bq/Kg), determined from the net peak areas of gamma spectrometer rays (604,7 KeV) through the leachates in 175 ml plastic container, V is the geometric volume of the geopolymer package (cm3), A0 is 134Cs activities incorporated initially in the package (Bq/Kg), S is the geometric surface area of the package (157.44 cm2), t is the leaching time (s).

## Results and discussion

### Characterization of IERs wastes

The grain structure characterization of the air-dried resin is performed using an optical microscope (DSX10-SXLOB Olympus Corporation, Tokyo, Japan). They are an equivalent mix of gel cation and a porous gel anion resins called Purolite NRW-3240, in the form of yellow translucent and clear amber spherical beads with diameters between 0.465 mm and 0.928 mm (**Fig.1**).



***FIG. 1****. Polarizing microscope images of used resin beads.*

The physical properties of the IER wastes are shown in **Table 3**. It was noticed that the increase in the percentage of IERs wastes resulted in an increase of conductivity due to the total ions leached out. Furthermore, the final pH has stabilized at a value of 4.16 that suitable to optimal immobilization of the resins. These used resin wastes are also characterized by higher water content (63.54 %) suggesting the hydrophilicity of the ionic polymer matrix (IERs). Moreover, IERs exhibited a different grain size distribution with high fine content.

TABLE. 3 The physical properties of IER with some physical characteristics

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Physical properties of IERs wastes** | Conductivity (µs/cm) | | ~ 2.8 | ~ 21.3 |  | ~ 33.2 |  | ~ 53.3 |
| pH | | ~ 5.74 | ~ 5.37 |  | ~ 4.97 |  | ~ 4.16 |
| Moisture (wt.%) | | ~ 63.54 |  |  |  |  |  |
| Grain size (mm) | D0.3 | | 0.4 |  |  |  |  |
| D0.4-1 | | 82 |  |  |  |  |
| D1.25 | | 5 |  |  |  |  |

### Chemical stability of the elaborated packages

**Fig.2** presents the visual appearance of packages immersed under DI water for 3 months. Packages prepared with the un-calcined UPS presented a better performance and desired features of durability after immersion test, in comparison to the packages prepared with MK (**Fig.2A**); this suggests that the presence of UPS particles were favourable for IERs retention under the **n**eutral nature. A possible reason for this could be that the UPS is actively involved in chemical reactions or a lower sensitivity under immersion ID water associated with its coarser particles. However, up to 50 wt.% of the un-calcined UPS, the packages exhibit higher amount of visual appearance heterogeneity contain relatively larger pores on the exterior surface; this was mostly ascribed to their higher amount of impurities and unreacted particles of the UPS.

Resuming the photograph of the packages with different concentration of the IER wastes, it appears that the addition of the resin beads has to positive influence on the durability indicators, while significant cracks and superficial deterioration were visually severe on the package with 15 wt.% (**Fig.2B**). As expected by visual examination, the introduction of the resin beads with different particle sizes as an addition to the geopolymer formulation allows reducing the apparent porosity of the elaborated package.



***FIG. 2.*** *Geopolymer packages after immersion in DI water for 3 months:* ***A****. effect of un-calcinated phosphate sludge (UPS),* ***B****. effect of Non-radioactive Resins (NR).*

### Compressive strength of solidified geopolymer packages

In order to assess the mechanical integrity of the elaborated packages, compressive strength experiments were performed at 7-days, 28-days and after immersion in DI water for 3 months (**Fig. 3 & 4**).

**Fig.3** shows the compressive strength results for the individual MK and binary MK-UPS geopolymer packages. The addition of un-calcined UPS promotes an increase in compressive strength by approximately 51% after 28 days compared to the reference package (MK/0PS-13RR). The increase of compressive strength is even greater after the immersion in DI water (3 months), (264%), which may be due to dual role of un-dissolved or partially dissolved UPS particles or bonding sites towards the neoformation of new reaction products such as (C,N,K)-A-S-H, C-S-H and C-A-S-H [18]. However, the excessive addition of UPS (50 wt.%) reduces the rate of chemical reaction (due to lower bending phases) and compressive strength, especially after immersion in DI water. This behaviour suggests that due to the incorporation of high amounts of UPS (lower amorphous phases) the extent of the geopolymerization reaction is lower. This can suggest an excessive amount of UPS leads to the formation of weak reaction products which can easily be leached out, damaging the chemical durability of packages as observed in **Fig. 2A**.



***FIG. 3****. Compressive strength of different individual and binary geopolymer packages. WAC:* ***waste acceptance criteria****.*

Although MK/30PS-13NR recorder higher strength (32.26MPa) than MK/42PS-13NR (31.55MPa), the latter was chosen as the optimal binary binder especially as the reduction in strength was limited to 5.60%. The appropriate selection was undertaken on the basis of two targets. The first is that replacing MK by UPS directly reduces consumption of alkaline activator while providing a suitable chemical environment to promote durability and resistance development. While the second target was to obtain higher amounts of reaction products as a result of an expected partial dissolution of calcite and dolomite when adding the UPS.



***FIG. 4****. Effect of non-radioactive resins addition (%) on compressive strength of binary geopolymer packages.* ***WAC****:* ***waste acceptance criteria****.*

**Fig. 4** shows the compressive strengths of the geopolymer packages with different loading ratio of IER wastes after 7, 28 days and after immersion ID water test. The package in the absence of solidified resin beads (Mk/42PS-0NR) was set as reference in this test (**Fig. 3**). Generally, the illustrated data revealed that the compressive strengths of package recorded a downward trend at different extents when the resin wastes loading were increased, with exception of the of the loading ratio of 15 wt.% of IERs. The highest strength values which met the waste acceptance criteria (WAC, [19]) were 36.14, 32.71 and 31.55 MPa and belonged to loading by 9, 11 and 13 wt.% resin beads. Therefore, 13 wt.% resin beads loading ratio of both MK/0PS-13RR and MK/42PS-13RR geopolymer packages was selected for leaching experiments.

### Leaching characteristics

The leaching performance of solidification packages is essential factor to characterize whether the packages effectively prevent the leaching of nuclide ions into the biosphere. As shown in **Fig.5a**, the leaching of 134Cs mostly occurred over the three days, with the leaching rate decreasing quickly over time. However, a small concentration of 134Cs also leached beyond day 7 in MK/0PS-13RR package without adding UPS. In contrast, very low amount Cs leaching was detected after 7days in MK/42PS-13RR package; moreover, the leaching rate values were maintained at very low concentrations in any timing of test during the entire leaching experiments.

Although Cs desorption was detected from days 3-7, but the total amount of leaching rate remained extremely low. This result indicates that the UPS-based geopolymer package has a good effect on immobilizing Cs which can satisfy the requirements for industrial applications. Further measurements under various conditions and analysis should be carried out to elucidate the [encapsulation](https://www.sciencedirect.com/topics/earth-and-planetary-sciences/encapsulation) mechanisms of Cs.

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***FIG. 5****. Effect of binder type on the leaching rate of 134Cs leached from solidified resin beads. (a) individual metakaolin (****MK/0PS-13RR****) and binary (****MK/42PS-13RR****) geopolymer packages and (b) Portland cement binder (****PCB-12RR****).*

Previous work from our laboratory have found that the Portland cement binder (PCB) can potentially use to treat radioactive resins loaded with 134Cs [16]. In the current work, the prepared package had a strength of 8.61MPa and was effective in encapsulating 12 wt.% of the spent resin over a wide range of cesium concentrations (with initial activity 107.93 Bq/Kg). As shown in **Fig.5b**, the highest leaching rate of 134Cs from PC-based packages occur on the 7 days of leaching. The leaching rates of Cs+ in the PCB decreased over time and dropped significantly before day 65. The leaching rate of Cs+ across 14 days reaches 2.66×10-8 cm/s for PC packages, and 1.58×10-09 cm/s for UPS-based geopolymer packages, respectively. This result showed that the immobilization ability of Cs+ in UPS-based geopolymer packages, with initial activity 257.98 Bq/Kg), is more outstanding than that of PCB packages. Therefore, UPS-based geopolymer package has clearly demonstrated its ability to improve the encapsulation performance of nuclear resins loaded with 134Cs as compared with individually MK-based geopolymer package and PC package.

### Environmental implications

The safe and rational use of greener nuclear energy substantially reduces fossil fuel consumption, reducing energy consumption and ensuring a long-term energy supply for human society. However, the safe elimination of nuclear wastes is an environmental and energy issue that the world must face and resolve. One way of solving this problem is to synthesis green materials by using industrial solid waste/mining wastes; these materials are then used to solidify nuclear waste. From a circular economy perspective, this approach enables the cost-effective and safe disposal of nuclear waste, while resolving the problem of high value for the use of solid waste. The developed UPS-based geopolymer package in this study demonstrated excellent solidification effects of radioactive grade resins toward large-scale utilization of phosphate industry by-products. Collectively, this paradigm provides an alternative pathway to achieve solidification of nuclear wastes and to treat industrial phosphate solid by-products in moving towards a circular economy model.

## Conclusions

The phosphate tailings-derived geopolymer package exhibited very effective encapsulation performance for radioactive grade resins loaded with cesium radionuclide. Non-radioactive resin beads could be solidified into MK/42PS geopolymer package by 9, 11 and 13 wt.% yielding 28-days compressive strength values (36.14, 32.71 and 31.55 MPa, respectively) greater than the waste acceptance criteria. The leaching rate values of cesium were two orders of magnitude lower than individual MK-based geopolymer package and the conventional PC package, indicating the better retention performance of the phosphate tailings-derived geopolymer package of radionuclide elements towards the biosphere.

The production of phosphate tailings-derived geopolymer package could achieve environmentally friendly character and large-scale resource use of phosphate industry by-products. This approach of handling nuclear waste with phosphate tailings allows for the cost-effective and safe disposal of nuclear wastes, while also promoting the high-value utilization of industrial solid wastes.

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