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IMMOBILIZATION OF SPENT ION EXCHANGE RESIN FROM PUSPATI TRIGA REACTOR USING FLY ASH-BASED GEOPOLYMER

(Pemegunan Resin Penukar Ion Terpakai dari Reaktor TRIGA PUSPATI Menggunakan Geopolimer Berasaskan Abu Terbang)

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Abstract

A laboratory study was conducted to evaluate the possibility of immobilizing spent ion exchange resins using geopolymer process that was prepared at room temperature. The main objective of this research is to determine the suitability of using geopolymer in radioactive waste immobilization. The spent ion exchange resins and fly ash were obtained from PUSPATI TRIGA Reactor and Sultan Salahuddin Abdul Aziz Power Plant, Kapar, Selangor respectively. The geopolymer was activated with NaOH and Na_2SO_3 at room temperature. Gamma spectrometry analysis indicated the presence of ^{40}K, ^{226}Ra and ^{232}Th in fly ash while in spent ion exchange resins showed the presence of activation and fission products i.e. ^{134}Cs, ^{137}Cs, ^{152}Eu, ^{54}Mn, ^{58}Co, ^{60}Co and ^{65}Zn. The composition of the materials that made up the monolith was 10% (wt) of spent ion exchange resin, with a compressive strength of 6.1 MPa. The leaching rates of the radionuclides were used as indicators of immobilization performance of the solidified waste forms. The leaching test shows different leaching rates of ^{134}Cs, ^{137}Cs, ^{226}Ra, ^{60}Co and ^{40}K from the monolith geopolymer. The results show that immobilization of spent ion exchange resins using fly ash based geopolymer significantly affect the leaching rate of the radionuclides. This finding could in future benefits the treatment of low level and intermediate waste generated from nuclear power plant operation.

Keywords: spent ion exchange resins, geopolymer, fly ash, radioactive waste

Abstrak

Kajian makmal telah dijalankan untuk menilai kemungkinan pemegunan resin penukar ion terpakai menggunakan proses geopolimer berasaskan abu terbang yang telah disediakan pada suhu bilik. Objektif utama kajian ini adalah untuk menentukan kesesuaian pemegunan sisa radioaktif menggunakan geopolimer. Resin penukar ion terpakai dan abu terbang diperolehi daripada Reaktor PUSPATI TRIGA (RTP) dan Loji Janakuasa Salahuddin Sultan Abdul Aziz, Kapar, Selangor. Geopolimer ini diaktifkan dengan campuran larutan NaOH dan Na_2SO_3 pada suhu bilik. Analisis spektrometri gama didalam abu terbang menunjukkan kehadiran ^{40}K, ^{226}Ra dan ^{232}Th manakala resin penukar ion terpakai menunjukkan kehadiran radionuklid pengaktifan dan pembelahan iaitu ^{134}Cs, ^{137}Cs, ^{152}Eu, ^{54}Mn, ^{58}Co, ^{60}Co dan ^{65}Zn. Komposisi bahan yang membentuk monolit geopolimer adalah 10 % (berat) daripada resin penukar ion terpakai, dengan kekuatan mampatan sebanyak 6.1 MPa. Kadar larut lesap jangka panjang bagi radionuklid digunakan sebagai petunjuk prestasi pemegunan sisa radioaktif. Ujian larut lesap jangka panjang dari monolith geopolimer menunjukkan kadar larut lesap yang berbeza bagi setiap radionuklid berikut: ^{134}Cs, ^{137}Cs, ^{226}Ra, ^{60}Co dan ^{40}K. Keputusan menunjukkan bahawa pemegunan resin penukar ion terpakai menggunakan geopolimer berasaskan abu terbang memberi kesan ketara kepada kadar larut lesap monolith geopolimer. Penemuan ini boleh memberi manfaat kepada rawatan sisa tahap rendah dan sederhana pada masa depan yang dijana daripada operasi loji kuasa nuklear.

Kata kunci: resin penukar ion terpakai, geopolimer, abu terbang, sisa radioaktif

Introduction

Davidovits was the first to introduce the term 'Geopolymer' in 1978 to describe a family of mineral binders with chemical composition similar to zeolites but have an amorphous microstructure. Al and Si are the critical components to this geopolymer. Geopolymer with amorphous microstructure will produce better strength with the presence of Al and Si, and is more soluble if prepared in an alkaline solution [1].

Geopolymer consists of a polymer framework Si-O-Al, with SiO_4 and AlO_4 tetrahedral are linked by sharing all the O_2 atoms. The presence of positive ions such as Na^+, K^+, Li^+ and Ca^+, in framework is necessary to balance the negative charged Al^{3+} in the coordination bond IV with oxygen. According to Davidovits [2] the empirical formula of these mineral polymers is shown in equation 1 below:

$$Mn\ [-(SiO_2)\ z\text{-}AlO_2]\ n.\ wH_2O \quad (1)$$

where as M is alkali elements or cations such as sodium or calcium, n is degree of polymerization, z = 1, 2, 3 or more and w is hydration degree

Geopolymer process involves three main steps [3] of a mixture of silica and aluminum sources and alkaline solution. It decomposes Al and Si atoms in the presence of hydroxide ions in solution. Second, the arrangement of ions occurs in solution, and finally the polycondensation reaction forming the 3D structure of silico-aluminate [4].

Ordinary Portland Cement (OPC) is a hydraulic binder that is commonly used in the conditioning of radioactive waste. According to Elimbi [5] and Wang [6] the production of OPC is a resource exhausting as it involves energy-intensive process that releases large amounts of greenhouse gases such as carbon dioxide into the atmosphere. Alternatively, fly ash can be used to replace Portland cement and fly ash is produced in electric power stations. By reusing this by product of electricity power generation station, it is expected to have impact on the total energy demand for producing concrete as well as the green house gas emissions into the atmosphere [7]. The Geopolymer Alliance [8] recommended that the rock-based geopolymer cements are ideal for environmental applications, such as the permanent encapsulation of radioactive or other hazardous wastes, toxic metals, as well as sealants, capping, barriers, and other structures necessary for remedying toxic waste containment sites. Figure 1 below shows fly ash collected from Sultan Salahuddin Abdul Aziz Power Plant, Kapar, Selangor.

Figure 1. Fly Ash from Sultan Salahuddin Abdul Aziz Power Plant, Kapar, Selangor

Radioactive waste in Malaysia is produced from various fields such as agriculture, medicine and industry. These activities generate radioactive waste, which occur either in gas, liquid or solid form [9]. Waste Technology Development Centre (WasTeC) of Nuclear Malaysia is given the responsibility to collect all institutional waste within the country for processing, treatment and storage. Untreated radioactive waste will cause problems and high

radiation exposure due to larger amounts of storage pending disposal. To overcome this problem, conditioning of radioactive waste should be done to facilitate their management. Therefore, this study is aimed to develop a method of conditioning the radioactive waste on a laboratory scale using fly ash-based geopolymer process and to study the leaching behavior of this radioactive waste.

Currently, WasTec is investigating treatment and disposal options for spent ion exchange resin. Spent ion exchange resin are considered to be problematic waste that in many cases requires special approaches and precautions during its immobilization to meet the acceptance criteria for disposal [10]. The spent ion exchange resins are generated from PUSPATI TRIGA Reactor (RTP), a TRIGA MARK II.TRIGA stands for Training, Research, Isotope Production from General Atomic. It is used for the purpose of training, research and isotope production. Figure 2 shows spent ion exchange resin used in this study.

Figure 2. Spent Ion Exchange Resin

Ion exchange resin used in the nuclear reactor normally will be used for 10 to 15 times. This depends on the exchange capacity and the regeneration efficiency. A new ion exchange resin will be used to replace the resin that has been contaminated with radioactive [11].The RTP produces approximately 50 kg per year of spent ion exchange resins [12]. A treatment and disposal method is needed due to continue generation of the spent ion exchange resins and limited storage capacity. Presently, large amounts of spent ion exchange resin are stored at WasTeC storage facility. Table 1 shows the spent ion exchange resin stored at WasTeC [13].

Table 1. Spent Ion Exchange Resin Stored at WasTeC

Year	Spent Ion Exchange Resin (kg)
2010	70
2008	30
2007	150
2006	20

WasTeC is in the process of immobilizing the spent ion exchange resins using geopolymer process. Geopolymers are produced by adding aluminosilicates to alkaline aqueous solution for dissolution and subsequent polymerization to form a solid [14]. Two main ingredient of geopolymers binder are source materials rich in silica and alumina, and alkaline liquids [3]. Immobilization of radioactive waste in accordance to IAEA [15] means a conversion of waste into stable waste form by solidification, embedding or encapsulation. The aim is to reduce the potential for migration of dispersion of radionuclides during handling, transport, storage and/or disposal. In the present work,

radioactive fly ash based geopolymer is investigated to determine its efficiency as cement-based materials and to evaluate the behavior of its addition on the leaching properties of the immobilized waste matrix.

Materials and Methods

Spent Resin Collection and Preparation

Spent ion exchange resin samples, generated from the water treatment process of TRIGA MARK-II were taken from the temporary storage facility located at WasTeC. The spent ion exchange resins were scooped and filled in clean 3 liter sealed plastic containers for characterization. Direct immobilization and encapsulation of spent ion exchangers was used in this study.

Fly Ash Collection and Preparation

The fly ash used in this study was obtained from Sultan Salahuddin Abdul Aziz Power Plant, Kapar Selangor. This power plant has been in operation since 1985. It contributes 23 % of the national electricity demand [16] and it uses only bituminous coal [17].

Design of Mix Proportion

The mix proportions are indicated in Table 2. This data is based on the work that has been carried out in which the parameters such as the ratio of fly ash to alkaline solution, the ratio of sodium silica (Na_2SiO_3) to sodium hydroxide (NaOH), NaOH molarity, volume of superplastersizer and waste loading has been considered to obtain the optimum value for each of these parameters. The ratio of fly ash / alkaline solution and Na_2SiO_3 / NaOH is 2.0 and 2.5 respectively. The materials were prepared according to the given ratio and mixed in the Hobart mixer for 2 minutes. The slurry was poured into a 50 mm x 50 mm x 50 mm stainless steel cube moulder and compacted. The samples were then vibrated to release any residual air bubbles. During hardening the geopolymer cement paste, the samples were covered with a thin film of polyethylene to avoid water evaporation and then kept for 24 h at ambient atmosphere of the laboratory before demoulding. Each sample was prepared in duplicates.

The alkaline solution used was a mixture of aqueous solution sodium hydroxide and sodium silicate. The alkaline activator used in this study is an analysis grade (AR). Sodium hydroxide (NaOH) and sodium silica (Na_2SiO_3) were obtained from Sigma Aldrich. NaOH is in the form of pellets while Na_2SiO_3 is in solution form. Solutions were stored for a minimum of 24 h prior to use to allow equilibration. To achieve higher workability, a commercially available superplasticizer supplied by Sika Kimia Sdn Bhd Malaysia was used.

Table 2. Details of Mix Proportion

Proportion	By Weight
Fly ash (%)	56
NaOH (12 M) (%)	8
Na_2SiO_3 (%)	20
Superplasterciser (%)	6
Spent Resin (%)	10

Analysis of Radionuclides Content: Spent resin

The spent resins and fresh ion exchange (as a control) samples were packed into 350 ml plastic container and measured using ORTEC hyper-pure germanium (HpGe) gamma spectrometer system with 30 % relative efficiency and a resolution of 1.74 keV at 1.33 MeV of ^{60}Co.The detector efficiency calibration was performed using a multinuclides standard source in 350 ml plastic container (containing certified concentrations of ^{241}Am, ^{109}Cd, ^{57}Co, ^{123m}Te, ^{51}Cr, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{88}Y and ^{60}Co), which was purchased from Isotopes Product Laboratories (IPL, USA). Gamma Vission analysis software was used to analyze the samples. The following radionuclides were searched for by gamma spectrometry based on the report of Omar et al.[18] regarding on radionuclides that were present in the

water cooler RTP is ^{24}Na, ^{42}K,^{51}Cr, ^{54}Mn, ^{56}Mn, ^{58}Co, ^{60}Co, ^{65}Zn, ^{76}As, ^{99}Mo, ^{99m}Tc, ^{122}Sb, ^{124}Sb, ^{131}I, ^{152}Eu, ^{187}W, ^{188}Re, ^{235}U, ^{239}Np, ^{59}Fe, ^{134}Cs and ^{137}Cs.

Fly ash

Wavelength Dispersive X-Ray Fluorescence (WDXRF) was used to study the chemical composition (wt. %) of fly ash. The fly ash samples were sent to Pusat Pengurusan Penyelidikan dan Instrumentasi (CRIM), Universiti Kebangsaan Malaysia (UKM) for chemical composition analysis.

Radioactivity of radionuclides in fly ash was also determined using gamma spectrometry technique. The fly ash samples were packed into 350 ml plastic container and measured using an ORTEC hyper-pure germanium (HpGe) gamma spectrometer system with 30 % relative efficiency and a resolution of 1.74 keV at 1.33 MeV of ^{60}Co. The detector efficiency calibration was performed using a multinuclides standard source in 350 ml plastic container (containing certified concentrations of ^{241}Am, ^{109}Cd, ^{57}Co, ^{123m} Te, ^{51}Cr, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{88}Y and ^{60}Co), which was purchased from Isotopes Product Laboratories (IPL, USA). Gamma Vission analysis software was used to analyse the samples. Gamma-ray energies of 1460 keV for ^{40}K, 1764.5 keV (^{214}Bi) and 351.9 keV (^{214}Pb) were use determine the concentration of ^{226}Ra. Gamma-ray energies of 911.2, 964.6 and 969.0 keV (^{228}Ac), were use determine the concentration of ^{232}Th.

Compressive Strength

Testing of the compressive strength of the sample was based on BS 1881-116:1993 standard. A total of 9 cube samples (50 mm x 50 mm x 50 mm) were prepared and tested at 3 different curing times (7, 14 and 28 days).

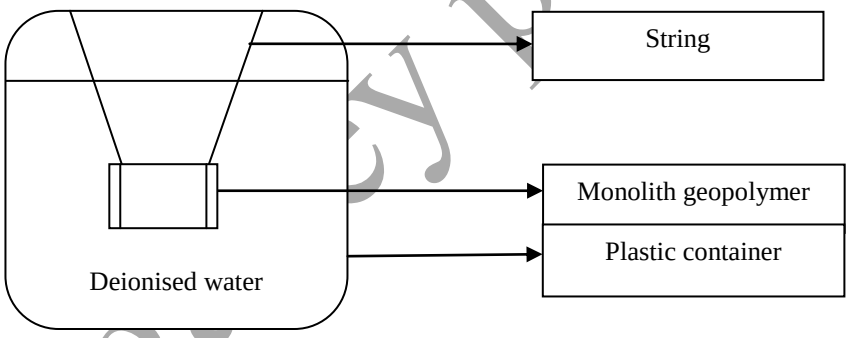

Figure 3. Diagrams on the Position of the Monolith Geopolymer Immersed In Deionised Water

Leaching Tests

Leaching test was carried out based on the ISO 6961 (1982) standard. The samples used for this purpose went through the process of control for 28 days, before the sample was placed in a plastic container. The amount of deionized water used and the dimensions of the container were as specified by ISO 6961 (1982). The changing frequency of the leachant shall be after 1, 3, 9, 14, and 30 days. Based on the standard ISO 6991 (1982), the dimensions of each plastic container must have a value of the ratio between 0.1 to 0.2 as shown in the equation 2 below:

$$R^i_n = \frac{a^i_n}{A^i_o F t_n} \ kgm^{-2}s^{-1} \quad (2)$$

R^i_n is the incremental leaching rate, in kg per m^2 per second for the i-th constituent; a^i_n is the radioactivity per second or weight in kg of each constituent leached during each leaching interval (Bq/s); A^i_o is the specific radioactivity, per second kilogram, or concentration in weight fraction initially present in the specimen ($Bqkg^{-1}s^{-1}$);

F is the exposed surface area of the specimen, in square meters (m^2); t_n is the duration of the n^{th} leaching period, in seconds (s); t is the cumulative leaching time in seconds; A^i_o and a^i_n are to be corrected for decay time and *n* is the time increment. Figure 3 shows the position of the monolith geopolymer in plastic container, immersed in deionized water as the leachate. The tests were run at room temperature.

Results and Discussion

Composition of Fly Ash

The chemical composition of fly ash determined by XRF is shown in Table 3. Based on the XRF result, it showed that the fly ash from Sultan Salahuddin Abdul Aziz Power Plant, Kapar Selangor was a class C fly ash due to the total percentage of SiO_2, Al_2O_3 and Fe_2O_3 of more than 50 % and SO_3 percentage content is less than 5%.

Table 3. Chemical Composition of Fly Ash

Oxide	(%) by mass	Requirements as per ASTM C618 Class C
SiO_2	44.39	-
Al_2O_3	19.90	-
Fe_2O_3	3.75	-
Total SiO_2, Al_2O_3 & Fe_2O_3	68.04	Min. 50%
CaO	5.44	-
SO_3	0.49	Max. 5%

Radionuclides content

The results of gamma spectrometric analysis of spent ion exchange resins, dried spent ion exchange resins and fly ash are shown in Table 4.

The result shows the highest activity concentration value for spent ion exchange resins was found for ^{60}Co (129725 ± 9567 Bq/kg) and the lowest was for ^{137}Cs (87± 2 Bq/kg) respectively. The results also show that fission products such as ^{134}Cs and ^{137}Cs were present in the spent ion exchanges, contradict to the expectation that it should contain only activation radionuclides. The presence of these fission products would indicate that there might be a cladding integrity loss [19-21] or pinhole of the fuel elements [22]. Detail assessment should be made to clarify these findings. While ^{152}Eu is primarily fission products, it could also be produced by neutron activation of nuclear reactor control rods [23]. The ^{54}Mn, ^{58}Co, ^{60}Co and ^{65}Zn were detected in the spent resin are activation product through (n,γ) reactions of either the corrosion product deposit on the fuel surface or the in-core structure materials [24]. As expected, the activity concentration of dried spent ion exchanges was higher than the wet spent ion exchanges. The highest activity concentration value for dried spent ion exchange resins was found for ^{60}Co (166015 ± 756 Bq/kg) and the lowest was for ^{58}Co (88 ± 4 Bq/kg).

The activity concentration of ^{40}K in the fly ash sample was found to be lower than the world average values of 420 Bq/kg [25]. While activity concentration of ^{232}Th and ^{226}Ra found to be higher than the world average values of 33 and 45 Bq/kg respectively. Factors that can influence the concentration differences for each country depend on the mineral content of coal and location of the mining [26].

Compressive strength

Figure 4 shows the compressive strength of the geopolymer samples with waste loading of 10 % spent resin. The highest compressive strength at day 28 was 6.1 MPa. According to IAEA [27], the minimum standards for compressive strength for monolithic of radioactive waste immobilization after reaching the age of 28 days was 320 - 7000 N/cm^2 or 3.2-70 MPa. Based on the result, the values obtained are within the recommended value by the IAEA.

Table 4. Radionuclides Identified In Spent Ion Exchange Resins and Fly Ash (Bq/kg)

Radionuclides	Half life	Activity Concentration (Bq/kg)		
		Spent ion exchange resins	Dried spent ion exchange resins	Fly Ash
^{152}Eu	13.33 y	621 ± 48	778 ± 156	-
^{137}Cs	30.17 y	87± 2	144 ± 0.1	-
^{134}Cs	2.06 y	1239 ± 64	1201 ± 114	-
^{65}Zn	243.8 d	10433.5± 594	3419 ± 123	-
^{60}Co	5.27 y	129725 ± 9567	166015 ± 756	-
^{58}Co	70.86 d	9834.3 ± 64	88 ± 4	-
^{54}Mn	312.29 d	77779 ± 5074	35003 ± 987	-
^{40}K	1.26 x10^{9} y	-	-	251.8± 9
^{226}Ra	1622 y	-	-	338.5± 9
^{232}Th	1.4 x 10^{10} y	-	-	422.7± 24

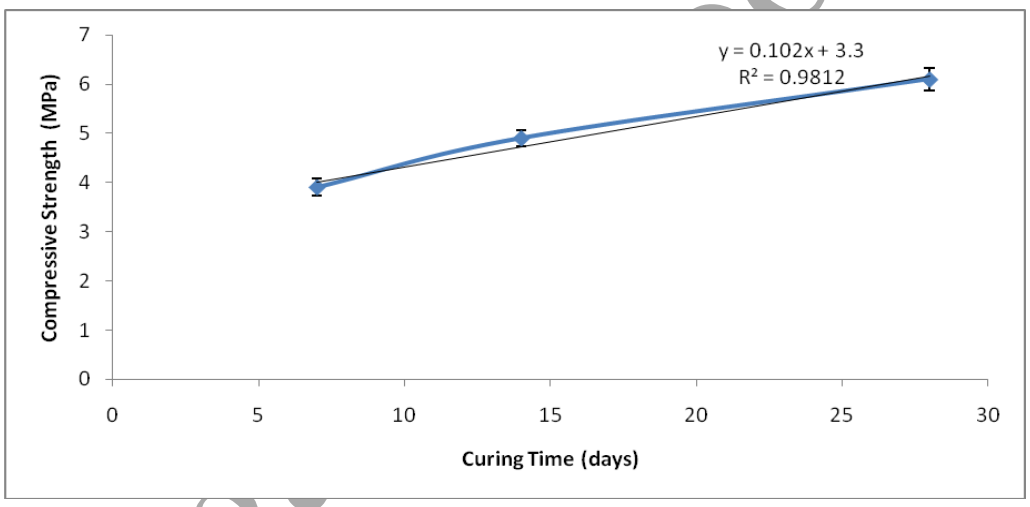

Figure 4. Compressive Strength with Waste Loading 10%

Leaching Test

Figure 5 shows the leaching rate of radionuclides from monolith geopolymer with 10% (wt) of spent ion exchange resin. The result showed that ^{134}Cs, ^{137}Cs, ^{226}Ra, ^{60}Co and ^{40}K were leached out from the monolith geopolymer during the laboratory leach test. The leaching rate of ^{40}K after the day 1 was the highest followed by ^{134}Cs, ^{137}Cs, ^{226}Ra and ^{60}Co respectively. The leaching rate of these radionuclides decreased over the 30 days of experiment as shown in Figure 3 except for ^{40}K, which increased after the days 15. Overall, the high leaching rate corresponds to 'swelling' of spent resin which produced micro-cracks or fractures in the entire monolith geopolymer. In addition, the spent resin used may react with the binder if there is a presence of water in the conditioning process [10]. Low concentrations of ^{134}Cs and ^{137}Cs were leached out from the monolith geopolymer. According to Perera et al. [28], cesium is very soluble in water and among the most difficult radionuclides to immobilize. Based on the leaching rate, it is predicted that the leaching percentage in 300 years (10 half-life of ^{137}Cs) and at the end of this period radioactivity will have decreased more [29]. It is expected the leachate of the samples will decrease when curing time is increased due to the increase of mechanical compressive strength of this geopolymer. For future research, it

is suggested that spent resin is treated before proceed for immobilization using geopolymer process such as pre-treatment processes for example dewatering or drying to get some reduction in total and to avoid swelling behavior of the spent resins during immobilization process.

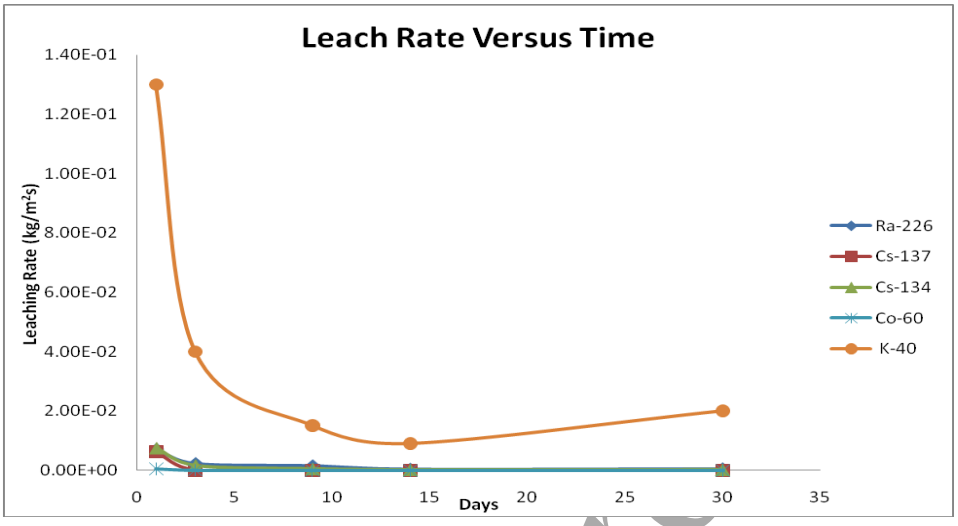

Figure 5. Leaching Rate versus Time

Conclusion

The results show that conditioning the spent resin together with fly ash at room temperature is able to produce geopolymer with reasonable strength properties. The composition of the materials that make up the monolith was 10 % (wt), with the monolith compressive strength of 6.1 MPa, well within the internationally acceptable value (320 - 7000 N/cm^2 or 3.2 - 70 MPa). Results from gamma spectrometry analysis showed the presence of activation products and fission products and leaching test shows that ^{134}Cs, ^{137}Cs, ^{226}Ra, ^{60}Co and ^{40}K were leached out from the monolith geopolymer.

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