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Modelling the Sorption of ^{152}Eu to Granitic Rocks and Sorption Verification Using Energy Dispersive X-Ray, Microanalysis

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Abstract

^{152}Eu is often used as a representative of trivalent actinides in the migration studies of radioactive nuclides in ground water systems. In the event of leached nuclides from a repository, humans have only the geomedia as the only retardation barrier by the process of sorption of leached radioactive radionuclides. In this study ^{152}Eu was used to study the sorption properties of different granitic rocks in batch sorption experiments and results modelled using non electrostatic correction models such as the Linear K_d, the Langmuir and the Freundlich isotherms. Sorption identification studies were carried out on Bulk granitic samples and analysed using Energy dispersive X-ray, microanalysis. Sorption verification studies of Eu sorption to a granitic bulk sample was conducted with the help of FIE QUANTA 600 Environmental scanning electron microscope, coupled with Oxford Instrument-INCA450 a solid state 2 diode type detector, operating at environmental pressure of <1 torr at the British Geological Survey. The micrographs and elemental spectra of the samples in $EuCl_3$ and DI water showed that sorption of Eu occurred at the different mineral phases of the granite sample with most of the sorption taking place at the mica phase. Taking into consideration the effect of surface area per gram of solid, results showed that the most sorbing among the granitic rocks was GrG while the least sorbing was RG. Based on the calculated R_d, sorption to the different granitic rocks studied can be ranked in the order; GrG > GG > GA > BG > RG. microanalysis showed sorption on the various component minerals of the granitic sample.

Keywords

Trivalent Actinides, Migration, Microanalysis

1. Introduction

Prediction of the retention mechanisms of radionuclides is a fundamental concern in the evaluation of the suitability of proposed sites for geological disposal/storage. In the study therefore of actinide retention, ^{152}Eu is used as a chemical analogue (Stumpf et al., 2002) for trivalent actinides such as Am(III) and Cm(III). It is important to note that in the study of radionuclides migration in the far field not only trivalent nuclides are important but mono and divalent radionuclides such as ^{63} Ni and ^{137}Cs. Lots of other studies on the migration of radio nuclides have been done such as work done by Ebong et al. (2025) Sorption experiments of Eu to different granitic minerals have been performed.

1.1. Sorption Models

1.1.1. Linear K_d Model

The partition (or distribution) coefficient, K_d, is a measure of sorption of contaminants to geomedia, and is defined as the ratio of the quantity of the adsorbate adsorbed per unit mass of solid to the amount of the adsorbate remaining in solution at equilibrium. K_d values are thermodynamically determined at stated equilibrium conditions as opposed to distribution ratios such as R_d which are not thermodynamically determined. In this work R_d is preferred to K_d. Values for K_d not only vary greatly between contaminants, but also vary as a function of aqueous and solid phase chemistry (Atomic Mass Data Center, 2007). Some adsorption studies are conducted in a systematic fashion to evaluate the effects of various parameters (such as pH, and ionic strength) on K_d. The results of a suite of experiments evaluating the effect of contaminant concentration on adsorption, while temperature is held constant, are called an "adsorption isotherm." Among all phenomena governing the mobility of substances in aqueous porous media and aquatic environments, the transfer of substances from a mobile phase (liquid or gaseous) to a solid phase is a universal phenomenon. That is the reason why the "isotherm", a curve describing the retention of a substance on a solid at various concentrations, is a major tool to describe and predict the mobility of this substance in the environment (Limousin et al., 2007) This isotherm often cannot of itself provide information about the type of reaction involved. For example, the retention can be either due to surface retention without creating three-dimensional structure or to precipitation of a new solid phase (Sparks, 2003; Veith & Sposito, 1977). However, isotherms give a general view of the distribution of radionuclides between the solid-liquid phases.

Isotherm models are used to describe the case where sorption relationships deviate from linearity. For many short-lived radionuclides, the mass present never reaches quantities large enough to start loading surface adsorption sites to the point that the linear K_d relationship is not applicable. However, long-lived radionuclides and stable elements can be found in leachates and groundwaters near waste sources at concentrations large enough to affect the saturation of surface adsorption sites.

The partition (or distribution) coefficient, K_d, is expressed mathematically as shown below, as the ratio of the quantity of the adsorbate adsorbed per unit mass of solid (Q) to the amount of the adsorbate remaining in solution at equilibrium (C).

$$K_d = \frac{Q}{C} \tag{1}$$

$$Q = \frac{V}{M}\left(C_{A0} - C\right) + Q_{a0} \tag{2}$$

Most of the time, the concentration of the compound retained on the solid Q is calculated by difference between the initial solute concentration C_{a0} and the final solute concentration C. In the case of retention stage, the solid concentration at equilibrium Q ($\text{mol}\cdot\text{g}^{-1}$) is given by Equation (2) with V being the volume of solution (dm^3), M is the solid mass (g) and Q_{a0} ($\text{mol}\cdot\text{g}^{-1}$) is the concentration of the compound initially retained by the solid, which must be measured or shown to be negligible (Limousin et al., 2007).

The use of a distribution coefficient in describing nuclide migration requires some assumptions:

1) The sorption process during migration is reversible.

2) The ratio of solute concentration between the solid and solution phases also remains constant (Chung-Kyun & Pil-Soo, 1999).

A more realistic approach to the concept of K_d, which is a thermodynamically determined value, is the R_d (Distribution ratio) of the solute between the solid and liquid phases, at the stated experimental conditions and it is not thermodynamically determined.

1.1.2. The Freundlich Isotherm

For rocks and minerals, contaminant adsorption can sometimes deviate from the linear relationship established by the distribution coefficient. In some circumstances, the amount of contaminant in solution contacting the solid will reach such a concentration that all adsorption sites would become saturated and the linear relationship between contaminant adsorbed to contaminant in solution would no longer hold.

Long-lived radionuclides and stable elements can be found in leachates and groundwaters near waste sources at concentrations large enough to affect the saturation of surface adsorption sites. The Freundlich equation; Equation (3), (Aksoyoglu, 1989; Atoniadis & Tsadilas, 2007) is one of the various models that have been employed for the study of metal adsorption. It expresses relation between the adsorbed quantity Q and the remained solute concentration.

$$Q = KC^{\frac{1}{n}} \tag{3}$$

The equation is expressed in the linear form as:

$$\text{Log}Q = \text{Log}K + \frac{1}{n}\text{Log}C \tag{4}$$

where Q is the concentration of metal sorbed ($\text{mol}\cdot\text{g}^{-1}$), C is the concentration of metal in the equilibrium solution ($\text{mol}\cdot\text{dm}^{-3}$), K ($\text{dm}^3\cdot\text{g}^{-1}$) and n (dimensionless) is a parameter that describes the heterogeneity of the sorption sites. A graph with log C as x-axis versus log Q as y-axis provides a line of slope $1/n$ and intercepts the y-axis at log K. According to the Freundlich equation, the isotherm does not reach a plateau as C increases. As $1/n$ tends to unity the surface becomes more uniform. Intact and crystalline minerals have higher $1/n$ values than pulverised and non-crystalline minerals. The constants are usually derived from a plot of sorbed concentration (Q) against concentration in solution (C). The Freundlich equation assumes that the surface of the solid is covered with a monolayer of sorbed species. The monolayer is not covered by any other layer. The Freundlich model does not account for finite adsorption capacity at high concentrations of solute (Ohnuki, 1994).

1.1.3. Langmuir Isotherm

Sorption by the Langmuir isotherm assumes the solid has a limited adsorption capacity $Q_{\max}$.

- Adsorption occurs up to the extent of one monolayer.
- All adsorption sites are identical.
- Occupation of a site is independent of the occupation of neighbouring site(s).
- The temperature is constant.
- The surface is uniform and homogeneous.
- The process is reversible.
- Each site retains one molecule of the given compound and
- All sites are energetically and sterically independent of the adsorbed quantity (Langmuir, 1918).

The reversibility/irreversibility of the sorption process is of fundamental importance for the understanding of the fate of radionuclides in the geological systems. If the process is reversible, the same isotherm should be valid for sorption and desorption under the same experimental conditions (Cui & Eriksen, 1997). The Langmuir Model equation takes the form as shown in Equation (5) (Soek, 2004):

$$Q = \frac{KbC}{1 + KC} \tag{5}$$

The linearised form of the equation is represented as

$$\frac{C}{Q} = \frac{1}{KB} + \frac{C}{B} \tag{6}$$

where b is the maximum adsorption capacity of the substrate ($\text{mol}\cdot\text{g}^{-1}$) and K is a constant representing the strength with which the solute is bound to the substrate ($dm^3 \cdot meq^{-1}$). Values of b and K can be determined by plotting the linearised Equation (6) (Kinniburgh, 1986).

The Freundlich and the Langmuir models have been used in describing results that showed deviations from a linear distribution. Empirical models like those mentioned are mathematical descriptions of the experimental data without any particular theoretical basis (US EPA, 1999).

The Langmuir model assumes that not all the adsorption sites are equally active; all adsorbing molecules do not exert an influence on their neighbourhood. Large molecules may occupy more than one adsorption site, and assuming that the adsorbed layer will be only one molecule thick is not valid. However, the Langmuir model gives us a basis for modelling adsorption by fitting data sets.

^{152}Eu, ^{154}Eu, and ^{155}Eu are produced primarily as fission products from fissile nuclides such as ^{235}U, ^{152}Eu can also be produced by neutron activation of nuclear reactor control rods.

$$^{151}\text{Eu} + 1n \rightarrow {}^{152}\text{Eu} \text{ neutron capture process}$$

^{151}Eu is a naturally occurring isotope and used in the control of fission reactions, due to its ability to accommodate neutrons. The associated gamma energies (in keV) and yields for ^{152}Eu are 121.78 (0.284), 244.7 (0.07), 344.28 (0.266), 778.91 (0.1296), 964.13 (0.143), 1085.8 (0.10), 1112.12 (0.1355), 1408.0 (0.2087).

Trace amounts of ^{152}Eu, ^{154}Eu, and ^{155}Eu are present in soil around the globe from radioactive fallout. They can also be present at certain nuclear facilities, such as reactors and spent fuel reprocessing plants. Europium is generally one of the more immobile radioactive metals in the environment. It preferentially adheres fairly strongly to soil (80). The importance of ^{152}Eu and ^{154}Eu is due to their relatively long half-lives for fission products, of 13.5 and 8.8 years, respectively. Due to behavioural similarities of the 4*f*-orbital lanthanides with some of the 5*f*-orbital actinides, Eu is often used as an analogue for the studies of trivalent actinides such as Am^{3+}, Cm^{3+} (Stipp et al., 2006).

1.2. XRD Results for the Different Granitic Rocks

The results of quantitative powder XRD analyses are summarised in **Table 1**. Powder X-ray diffraction analysis indicated that the five granites had approximately similar mineralogies and were predominantly composed of quartz (mean ca.33%), plagioclase feldspar (mean ca.31%) and K-feldspar (mean ca.31%) together with small/trace amounts of “mica” (undifferentiated mica species possibly including muscovite, biotite, illite, illite/smectite etc.). Small amounts of amphibole were also identified in the samples “Biotite granite” and “Rapakivi granite”. Traces of chlorite, kaolinite and smectite were also identified in some of the samples.

Further elemental characterisation and identification of the granite samples were carried out using energy dispersive spectroscopy. The elemental composition of elements present confirmed samples to be granites.

Table 1. Summary of quantitative whole-rock XRD analysis.

	Mineralogical percentage composition							
sample	amphibole	smectite	chlorite	kaolinite	K-feldspar	“mica”	plagioclase	quartz
Graphic Granite	nd	nd	nd	<0.5	49.4	0.5	21.6	28.3
Granite Adamellite	nd	<0.5	<0.5	nd	32.9	3.1	25.7	38.1
Biotite Granite	2.9	<0.5	<0.5	nd	17.2	7.4	40.0	28.1
Grey Granite	nd	nd	<0.5	nd	22.6	4.3	34.4	38.6
Rapakivi Granite	3.5	<0.5	<0.5	<0.5	32.1	1.6	29.2	33.3

nd = not detected, “mica” = undifferentiated mica species including muscovite, biotite, illite and illite/smectite etc (Wagner & Kemp, 2011).

2. Experimental

^{152}Eu spike solutions were prepared from dilutions of an initial 1 cm^3 of 37 MBq stock. The diluted solutions provided experimental stock giving final gamma counts of approximately 1000 counts per second in each experimental sample. Counting was performed using a Cobra II Auto Gamma within an energy range of 0 to 1500 keV, without the using cocktail. The above procedure was repeated as necessary, based on experimental requirements. As Eu has a life of 4933 years, decay corrections was not required, over the comparatively short experimental period.

Background corrections to the measured counts were made by counting blank samples without added radioactivity. The measured value for the blank sample (sample without added radioactivity mixed with liquid scintillation cocktail) is then subtracted from the measured counts of the sample. Corrections for wall and filter sorption were made by washing the filters and vials with nitric acid. The solution with the leached metal was counted. The results obtained from filter sorption were less than 1 % of the total counts of the sample.

The adsorbents were first reduced in size. The size reduction was necessary for the performance of batch equilibrium experiments because sorption capacity is proportional to the total surface area available and the total surface area of non-porous particles is inversely proportional to the particle diameter (Aksoyoglu, 1989). In addition, the kinetics of processes controlled by diffusion in porous particles is directly related to particle size. Smaller adsorbents will therefore require shorter equilibration times if any porosity was present (Aksoyoglu, 1989; Murali & Mathur, 2002) Samples were crushed and pulverised using a ball mill and sieved to obtain a particle size range of 46 to 250 µm. 0.2 g of the pulverised samples were mixed with 40 cm^3 of non-active research-grade $EuCl_3$ solution (Aldrich), giving a solid-liquid ratio of 1:200. Experiments with ^{152}Eu were analysed using the Cobra (II) Auto Gamma counting between 100 to 1500 keV at 2 sigma. 1 x After equilibrating for the required time, two cm^3 of the supernatant were removed and counted. Blank samples with no solids were also prepared. All experiments samples were carried out in triplicates and mean values used in processing the data used in the isotherm. The counts per vial for each sample did not vary by more than 1 percent.

3. Results and Discussions

3.1. ^{152}Eu Sorption to Granitic Rocks

Eu is one of the 14 elements in the Lanthanide series used as analogues for the trivalent actinides and exhibits a strong sorption on mineral surfaces (Hsieh et al., 2024; Molodtsov, 2019) Fitting the results to different sorption models, showed that sorption was different from one granitic rock to another. Best fit models showed that sorption was best described by the Langmuir model for GG, Linear K_d model for GA, BG and GrG, and by the Freundlich model for RG. Sorption parameters are shown in **Table 2**. The difference in sorption models is reflected in the R_d values calculated. R_d values for GA, BG and GrG obtained from the linear sorption isotherms were in the range 20 to 50 $cm^3 \cdot g^{-1}$ for the granitic rocks. Allard et al., and Erdal et al., (Allard et al., 1979; Erdal et al., 1979) obtained K_d value of 8 - 32 $m^3 \cdot kg^{-1}$ and values of around 0.24 and 0.55 $m^3 \cdot kg^{-1}$ for Eu sorption to Finnsjön granite respectively. Taking into consideration the effect of surface area per gram of solid, results showed that the most sorbing among the granitic rocks was GrG while the least sorbing was RG. Based on the calculated R_d, sorption to the different granitic rocks studied can be ranked in the order; GrG > GG > GA > BG > RG.

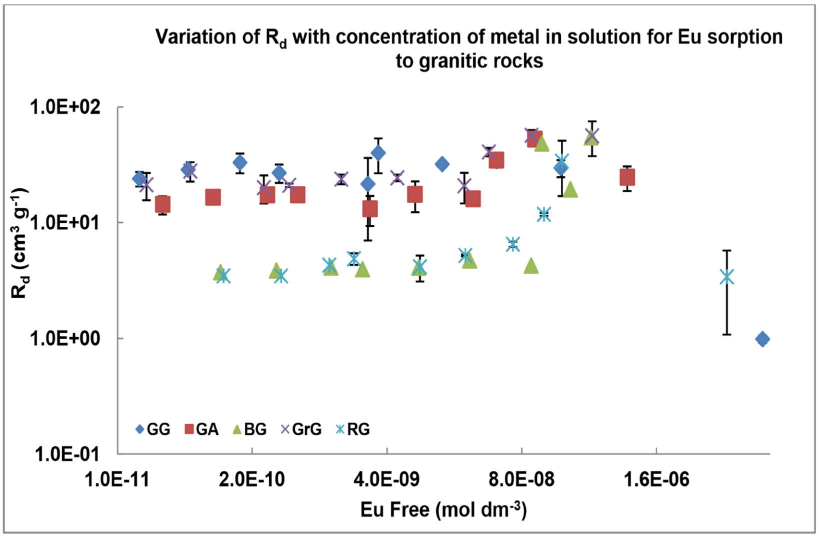

Figure 1. Variation of R$_d$ with metal loading for different granitic rocks used in sorption experiments.

Figure 1 shows the sorption isotherms for the different granitic rocks. From this, it is evident that sorption occurred linearly for all the samples up to about 1×10^{-7} $mol \cdot dm^{-3}$ of Eu loading. Above this concentration range, sorption deviated from linearity as a result of saturation/modification of sorption sites. The deviation from linearity is highlighted in **Figure 1**. According to Giles et al. (1974), deviation from linearity occurs in adsorption where, individual solute molecules bound to the solid interact with each other. This increases the strength of the individual solute bonds to the solid surface when the solid has low contaminant loading. Thus, for a brief period during adsorption, the first bound molecules enhance adsorption of the next molecules that bind to the solid. From **Figure 1**, deviation from linearity is evident from the decrease in R_d with increasing, metal concentration in solution.

Values of N close to unity RG implied that all sorption sites were energetically identical, and sorption occurred by a single mechanism. When N is close to 1, the Linear K_d sorption model and the Freundlich models are equivalent, as such deviation from linearity does not occur. **Table 2** shows the different sorption parameters studied and the best fit models that are used in describing the sorption process.

Table 2. Sorption parameters for Eu sorption to granitic rocks and minerals. Data in table determined by fitting experimental to the linearised Freundlich isotherm, linearised Langmuir and the Linear model. Only best fit models are shown on the table, $^*R_d$ is the arithmetic mean of the R_ds derived from individual points. R_{d^*} is the mean R_d corrected for surface area per gram effect. N is a dimensionless factor related to the heterogeneity of the sorption sites.

		Langmuir model		Freundlich	Linear		
		Granitic rocks					
	B (mol·g^{-1})	K (d·m^{-3} meq)	$^*R_d$ (cm^3·g^{-1})	N	R_d (cm^3·g^{-1})	R_{d^*} (cm^3·g^{-1}·m^{-2})	BF
GG	1.73×10^{-5}	1.9×10^6	27 ± 5.2			9.6 ± 1.8	L
GA					24	7.9 ± 1.2	Li
BG			15 ± 5.3		15.1	5.3 ± 1.9	Li
GrG					52.1	11 ± 2	Li
RG			8.1 ± 2.2	1.13		2.9 ± 0.8	F

Graphic Granite GG, Biotite Granite BG, Granite Adamellite GA, Rapakivi Granite RG, Grey Granite GrG. BF = Best fit model, B = Maximum sorbable amount for the Langmuir model, K = Langmuir parameter, relates to the binding strength, $^*R_d$ = mean R_d, L, F, Li, stand for Langmuir, Freundlich, Linear and models respectively. BM = Best fit model, N = The Freundlich parameter, relates to the heterogeneity of the sorption sites, indicative to the presence of different sorption mechanisms.

3.2. Sorption Verification Using Energy Dispersive Microanalysis

Sorption verification studies of Eu on granitic bulk sample was conducted with the help of FIE QUANTA 600 Environmental scanning electron microscope, coupled with Oxford Instrument-INCA450 a solid state 2 diode type detector, operating at environmental pressure of <1 torr at the British Geological Survey. The micrographs and elemental spectra of the samples in $EuCl_3$ and DI water showed that adsorption of Eu occurred at the different mineral phases of the granite sample with most of the sorption taking place at the mica phase **Figure 5**. The attribution of higher sorption capacity to mica solely based on the microanalysis is not fully justified. While mica might be a significant sorption site, the contribution of other minerals cannot be ruled out. Attribution of high sorption values to mica have also been highlighted by work done by Ebong et al., (2025) in which they studied how ^{63}Ni sorbed to different granitic components.

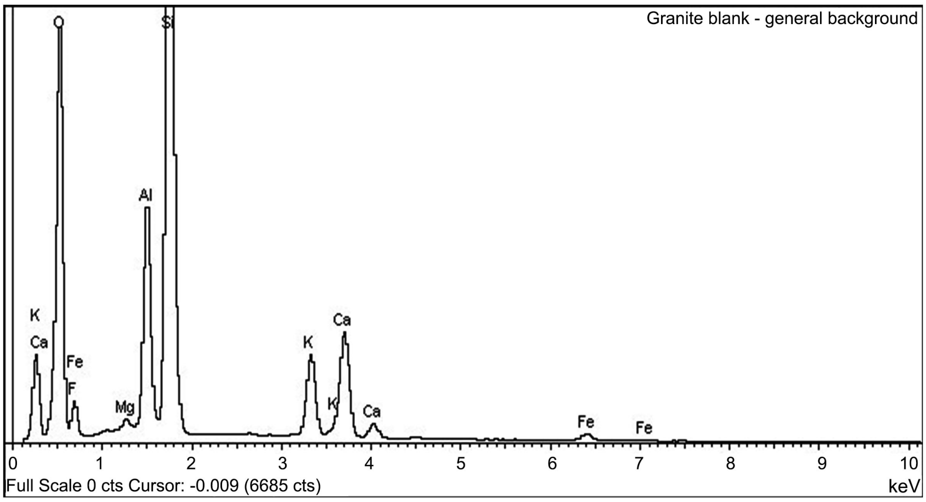

Figure 2. Energy dispersive X-ray, microanalysis of blank granite sample in DI, using a wavelength of 700 nm, a solid state 2 diode type detector for the back scatter electrons in a low-pressure mode (0.98 torr of water vapour), and a specimen current of 0.98 nA. Figure shows the elemental composition of granite, with high concentrations of Si and O.

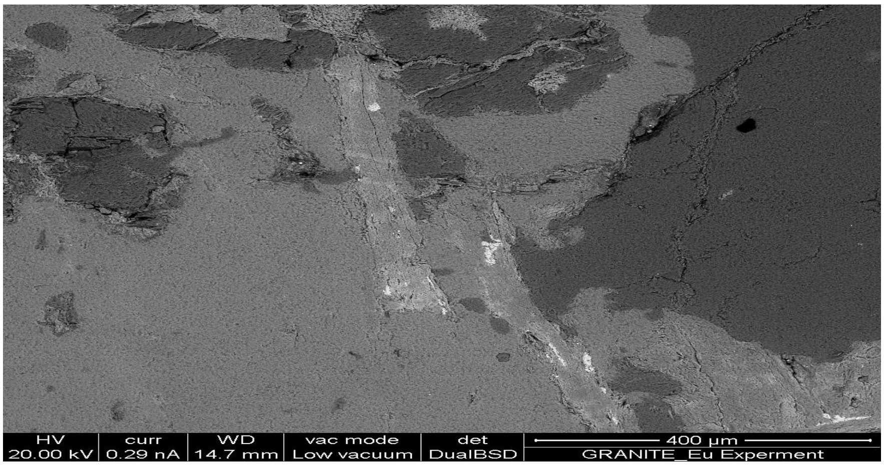

Figure 3. Energy dispersive X-ray Micrograph of granite sample doped with Eu, showing sorption of Eu (White spots on different areas of the granite sample).

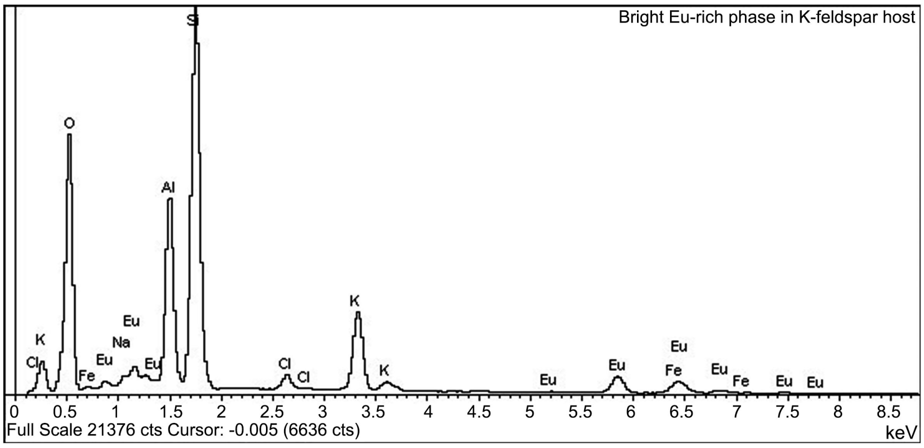

Figure 4. Energy dispersive X-ray, microanalysis of feldspar showing Eu peaks.

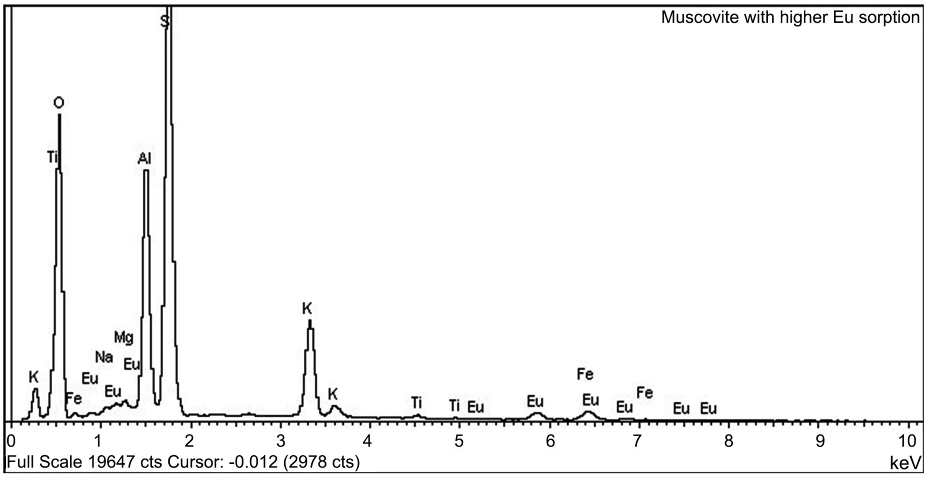

Figure 5. Energy dispersive X-ray, microanalysis of mica showing Eu peaks.

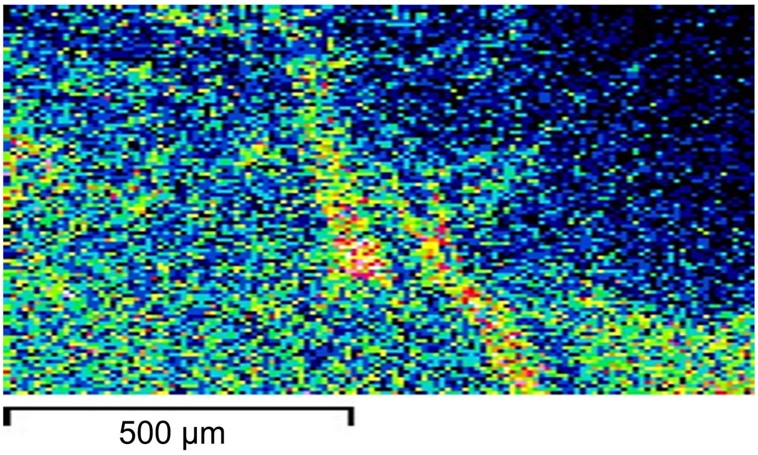

Figure 6. Energy dispersive X-ray, microanalysis map, showing elemental mapping of Eu on the granite surface. Micrograph shows high concentration of Eu on the mica surface.

4. Conclusion

Non electrostatic correction models were applied to the batch sorption data and results showed that sorption occurred by different sorption mechanism with GA, BG and GrG fitting best to the Linear K_d model as shown in **Table 2**, while GG and RG fitted to the Langmuir and Freundlich respectively. sorption capacity of Eu to granitic materials as shown recently in by Palágyi et al. (Palágyi et al., 2009). Very strong sorbing materials such as granitic rocks have been shown to have very low deviation from linearity hence, the high retention capacities of these rocks. Correcting the calculated R_d values for effective surface area showed clearly that sorption varies with the surface area available for sorption. Results for sorption verification using Energy dispersive X-ray, microanalysis, showed that sorption took place mostly on the major constituents of the granitic rock. **Figure 6**, (Energy dispersive X-ray, microanalysis of blank granite sample in DI, using a wavelength of 700 nm, a solid state 2 diode type detector for the back scatter electrons in a low-pressure mode (0.98 torr of water vapour), and a specimen current of 0.98

nA.) shows the elemental composition of granite, with high concentrations of Si and O, as blank sample. **Figure 3** and **Figure 6** are bulk sample of granite showing sorption sites for ^{152}Eu. Further microanalysis showed sorption on the various component minerals of the granitic sample **Figure 4** and **Figure 5**. It is thus left to investigate the component additive model for the sorption process. This work was thus to carry out sorption of Eu to various granitic rocks and verify the sorption process took place. The only safety barrier from the engineered barrier is the far-field which is the geological media close to the near-field, In the situation where radionuclides are leached from the near field, the far field is expected to have substantial retentive properties to retard the radionuclides from reaching the ground water. This study like many others showed that granitic rocks have a high retention ability and serve as a potential deep underground burial site.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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