# DISPERSION AND SORPTION CHARACTERISTICS OF URANIUM IN THE ZEOLITE-QUARTZ MIXTURE AS BACKFILL MATERIAL IN THE RADIOACTIVE WASTE REPOSITORY

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## ABSTRACT

The experiment of sorption and dispersion characteristics of uranium in the zeolite-quartz mixture as candidate of raw material of backfill material in the radioactive waste repository has been performed. The objective is to know the effect of zeolite and quartz grain size on the zeolite-to-quartz weight ratio that gives porosity ( $\varepsilon$ ), permeability (K), and dispersivity ( $\alpha$ ) of uranium in the zeolite-quartz mixture as backfill material. The experiment was carried out by fixed bed method in the column filled by the zeolite-quartz mixture with zeolite-to-quartz weight percent ratio of 100/0, 80/20, 60/40, 40/60, 20/80, 0/100 wt. % in the water saturated condition flowed by uranyl nitrate solution of 500 ppm concentration ( $C_o$ ) as uranium simulation which was leached from immobilized radioactive waste in the repository. The concentration of uranium in the effluents represented as  $C_t$  were analyzed by spectrophotometer Corning Colorimeter 253 every 15 minutes, then using  $C_o$  and  $C_t$  uranium dispersivity ( $\alpha$ ) in the backfill material was determined. The experiment data shown that 0.196 mm particle size of zeolite and 0.116 mm particle size of quartz on the zeolite-to-quartz weight ratio of 60/40 wt. % with  $\varepsilon = 0.678$ , K =  $3.345 \times 10^{-4}$  cm/second, and  $\alpha = 0.759$  cm can be proposed as candidate of raw material of backfill material in the radioactive waste repository.

Keywords: backfill material, quartz, radioactive waste, zeolite.

### INTRODUCTION

A final repository for small and medium activities of the radioactive waste recomended by International Atomic Energy Agency e.g. can be done by near surface disposal and shallow land burial in the fine repository building at the best location. Final repository must have multibarrier system consisting of engineered barriers and natural barriers such as shown in Fig 1.

Backfill material is a segment of engineered barriers, it is a porous solid material from natural minerals that can be used to retard the radionuclides migration from the immobilized radioactive waste in the repository by which the environmental exposure on the soils as natural barriers in the surrounding repository can be reduced.

As backfill materials usually used natural minerals with the condition as follows : very low pore to minimize groundwater flow and ionic diffusion, good sorption properties to retard the movement of radionuclides in case the container is broken, sufficiently high thermal conductivity to provide adequate heat dissipation to the surrounding rock, sufficient load-bearing capacity to be supported as the container but sufficiently plastic to dissipate high localized stresses, long-term chemical and physical stability under the temperatures, pressures and groundwater conditions of the vault [1].

The factors affecting on the migration of radionuclides from radioactive waste repository to surrounding soils are rate of container failure, porous medium effects, waste-form release processes, molecular diffusion, sorption and precipitation, nuclear decay, and vault-geosphere boundary condition [2].



Fig 1. Repository model of radioactive waste by shallow land burial system

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Scientists such as Lopez, LeNeveu, Aoki, Miyahara, Conca, [1, 2, 3, 4, 5] have already conducted the experiment on engineered barriers using local mineral e.g. zeolite, clay and quartz as backfill material and buffer material as part of engineered barriers system in the radioactive waste repository.

According to Lopez [1], the use of quartz as the backfill material from the mixture of bentonite-quartz to radioactive waste repository in Sweden is 50 wt. % of bentonite-quartz. Backfill materials composition that has been studied by other scientist contain e.g. 52 wt. % quartz, 25 wt. % montmorillonite, 14 wt. % vermicullite, 4 wt. % magnetite, 2 wt. % apatite, 2 wt. % atapulgite [6].

Particle size of minerals affects the physical characteristics i.e. bulk density ( $\rho_b$ ), effective porosity ( $\epsilon$ ), and permeability (K). Physical characteristics of mineral, chemical-physical incident such as adsorption and ion exchange in the backfill material influence on the importance of the retardation of radionuclides migration [7]. According to LeNeveu [2], dispersion of radionuclides dissolved in the solvent flown through the pore solid medium was expressed by dispersivity.

In this paper physical and dispersion characteristics of the zeolite-quartz mixture as candidate of raw material of backfill material in the radioactive waste repository is reported.

### EXPERIMENTAL SECTION

#### **Preparation of Zeolite and Quartz Powders**

Zeolite from Sampang Patuk Gunung Kidul have mineral composition : clinoptilolite (24,64%) and modernite (59,24%). Whereas chemical composition as follows : SiO<sub>2</sub> (64,74 – 66,59%), Al<sub>2</sub>O<sub>3</sub> (13,89 - 14,17%), Fe<sub>2</sub>O<sub>3</sub> (0,96 – 1,54%), CaO (1,64 - 2,81%), MgO (0,60 – 0,94%), Na<sub>2</sub>O (1,23 - 1,47%), K<sub>2</sub>O (0,95 – 1,27%), MnO (0,16 – 0,18%), H<sub>2</sub>O (0,22 – 2,61%) [8].

Gravel of zeolite was dried in a oven until its constant weight, then it was crushed to powders. The zeolite powders was poured in the sieving pan of ASTM ISO 585-R20 standard with the sieve from upper to bottom of 16 mesh and 30 mesh, and then were sieved. The zeolite powder grain size of -16+30 mesh was kept on the sieving pan of 30 mesh. The same method was used for the sieve from upper to bottom of 30 and 40 mesh, 40 and 50 mesh, 50 and 60 mesh, 60 and 70 mesh, 70 and 80 mesh respectively until the grains size zeolite of -30+40 mesh, -40+50 mesh, -50+60 mesh, -60+70 mesh, -70+80 mesh respectively were obtained. The grain size of zeolite that passes and remains on the sieve can be expressed by particle diameter, then particle diameter is determined by an equation as follows [9] :

$$d_{p} = \sqrt[3]{\left[\frac{(d_{1}+d_{2})((d_{1})^{2}+(d_{2})^{2})}{4}\right]}$$
(1)

with  $d_p$  = average particle diameter,  $\mu m$ 

 $\textbf{d}_{_1}$  = particles diameter pass the sieve,  $\mu m$ 

 $d_2$  = particles diameter remain on the sieve,  $\mu$ m. Preparation of quartz powders was carried out by the same method as that of zeolite powders.

### Determination of Bulk Density of Zeolite and Quartz Powders by Method ASTM D1895B A21.A

Zeolite powders were poured excessively into the bowl of cylindrical glass with known volume (Vb) and weight (Mk). The excess volume of zeolite powders were scraped horizontal by using thin blade precise at upper segment of the bowl. The bowl filled with zeolite powders weighed as Mb. Zeolite bulk density ( $\rho_b$ ) is determined by equation :

 $\rho_{b} = \frac{Mb - Mk}{Vb}$ (2)

# Determination of Effective Porosity of Zeolite and Quartz Bed

The porosity of samples was determined by an apparatus as shown in Fig 2. Glass column 2 filled with the powder of local mineral (zeolite or quartz) of  $V_u$  bed volume was flowed with aquadest from burette 1 through bottom of glass column 2. When the aquadest stream is precisely at bed bottom of local mineral powder at C boundary on the glass column 2, then valve K on burette 1 was closed. Noted volume at A boundary, then aquadest was flown to glass column 2 filled with the local mineral powder by opening K valve.



Fig 2. Apparatus for effective porosity determination



Fig 3. Apparatus for permeability determination by constant head method

After all of the local mineral powder in the glass column 2 was flown with aquadest to D boundary, then K valve was closed. The decrease of aquadest volume in the burette from A to B boundary as was noted  $V_{\rm p}$  that represents particle pores and cavities between particles of local mineral powder in the glass column 2. According to LeNeveu [2] and Ven Te Chow [10], effective porosity ( $\epsilon$ ) can be calculated by equation as follows :

$$\varepsilon = V_p / V_u \tag{3}$$

# Determination of Permeability (K) of Zeolite and Quartz Bed by Constant Head Method

The porosity of samples was determined as shown in Fig. 3. Vertical glass column C with inner diameter of d = 1.75 cm filled with local mineral powder with height of L = 10 cm. Feeder glass column 1 filled with aquadest on the head loss of H = 50 cm. Distilled water from reservoir 1 was flown to the column 2 through the bottom section. As the steady condition was achieved i.e. stream of inlet = stream of outlet column C, the stream of aquadest was stoped by closing the outer valve of reservoir 1. The determination of time of aquadest stream (t) was began until the effluent volume of V = 5  $\text{cm}^3$  by opening the outer value of reservoir 1. Permeability can be determined by constant head method by equation as follows [10] :

$$K = \frac{V.L}{A \dagger H}$$
(4)

with V = volume of effluent in t time (cm<sup>3</sup>), L = height of local mineral bed in the glass column (cm), A = cross area of glass column (cm<sup>2</sup>), t = time of V volume (second), H = head loss (cm).



**Fig 4.** Apparatus for uranium dispersion and sorption determination in the backfill material

### Determination of Dispersivity and Distribution Coefficient of Uranium in the Zeolite-Quartz Mixture as Backfill Materials

The determination of dispersivity was carried out using an apparatus shown in Fig. 4 with a column filled with samples of zeolite-quartz mixture as simulates of backfill materials saturated with distilled water. Distilled water under samples of zeolite-quartz inside the column will exit when the distilled water was opened. As the distilled water surface is precisely reaching precission the surface of zeolite-quartz samples inside the column, influent from reservoir 1 (as simulated the uranium leached due to the infiltration of surface water which pass the radioactive waste repository) flows through the column. The time by which the influent flows was recorded. The effluent from the column was collected stored in the graduated cylinder glass every 15 minutes, then volume was measured. The effluent of 1 cm<sup>3</sup> was put in the measuring cylinder glass of 5 0.5 cm<sup>3</sup> of arsenazzo was added, cm<sup>3</sup>, at which followed by the addition of 4.5 cm<sup>3</sup> HNO<sub>3</sub> 3M. It was then shaked homogenizely for 15 minutes in order the reaction completes. After that uranium concentration in the effluent was analyzed using spectrophotometer Corning Colorimeter 253 at optimum wave length.

Interaction between nuclides and absorbent might be caused by chemical and physical interaction. One is interrelated to the other and it could not be defined separately. This basic phenomenon is unknown by specific model. Those interaction could empirically described by distribution coefficient ( $K_d$ ). According to Lopez [1] and Champ [11], distribution coefficient is a constant volue to express the ratio of total of chemical species adsorbed in the solid to concentration of chemical species in the solution contacted with solid at steady state. Distribution coefficient at batch process is determined by equation as follows [12]:

$$K_{d} = \frac{(C_{0} - C_{e})/W}{C_{e}/V_{e}}$$
(5)

with  $K_d$  = distribution coefficient (cm<sup>3</sup>/g),  $C_o$  = concentration of solute in the influent (g/cm<sup>3</sup>),  $C_e$  = concentration of solute in the effluent (g/cm<sup>3</sup>), w = weight of absorbent (g),  $V_e$  = volume of effluent (cm<sup>3</sup>).

According to Poernomo [9], the determination of distribution coefficient at continuous flow process in the column filled with adsorbent of the porous solid medium is expressed by equation as follows :

$$\kappa_{d} = \frac{V_{T}.C_{o} - \Sigma V_{t}.C_{t}}{\Sigma V_{t}.C_{t}} \cdot \frac{V_{T}}{w}$$
(6)

with V<sub>T</sub> = volume at the time where C<sub>t</sub> = C<sub>o</sub> (cm<sup>3</sup>), C<sub>o</sub> = concentration of uranium in the influent ( $\mu$ Ci/cm<sup>3</sup>),  $\Sigma$ V<sub>t</sub>.Ct = accumulation of uranium concentration in the effluent where C<sub>t</sub> = C<sub>o</sub> ( $\mu$ Ci), w = weight of absorbent bed in the column as backfill material simulation (g).

Weight of absorbent bed in the column is determined by equation as follows :

 $w = \rho_b \cdot v_b$  (7) with  $\rho_b$  = bulk density of absorbent bed (g/cm<sup>3</sup>),  $v_b$  = volume of absorbent bed (cm<sup>3</sup>) expressed by equation  $v_b = (\pi/4) \cdot d^2 \cdot L$ , which d = diameter of column (cm), L = height of absorbent bed in the column (cm).

According to Guven [13], dispersivity can be determined by equation :

$$\alpha = \frac{3.L}{16.\pi} \frac{\left(t_{1,0} - t_{0,0}\right)^2}{\left(t_{0,5}\right)^2}$$
(8)

with L = height of absorbent bed in the column (cm),  $t_{1,0}$  = time at condition solute concentration in the effluent  $C_o$  =  $C_t$  (minutes),  $t_{0,5}$  = time at condition Ct/Co = 0.5 (minutes),  $t_{0,0}$  = residence time of solution in the absorbent bed in the column (minutes), and  $t_{o,o}$  is calculated by equation as follows :



Figure 5. Effect of grain size on bulk density

$$t_{0,0} = \frac{L.A}{Q.\epsilon} = \frac{\pi.d^2.L}{4.Q.\epsilon}$$
(9)

with L = height of absorbent bed in the column (cm), A = cross area of glass column (cm<sup>2</sup>), Q = volumetric velocity of influent (cm<sup>2</sup>/minute),  $\varepsilon$  = effective porosity of absorbent bed in the column, d = inner diameter of glass column (cm).

#### **RESULT AND DISCUSSION**

The grain size affects the physical characteristics of zeolite and quartz, therefore by knowing the grain size the best physical properties will be determined. Physical characteristics e.g. bulk density ( $\rho_b$ ), effective porosity ( $\epsilon$ ), and permeability (K) were shown in Fig 5, 6, and 7 respectively.

Fig. 5 shows that the smaller the grain size of the samples, the higher its density. This case will be interesting because at constant volume; the smaller the grain size, the greater total particles to fill its volume. As the total of particles increases, the samples weight will increase theat lead to the increase of the bulk density if it is determined by equation (2). The greater number of total particles, the larger bulk density determined by using equation (2) due to the more weight the samples.

From Fig. 6, is shown that the smaller of grain size of samples, the greater the porosity ( $\varepsilon$ ). This case will be interesting, because grain size smaller in the constant bed volume, then total of particles will be larger. Every particle has internal pore, and then particles were fill bed volume to form external pore between particles. Powders of the backfill material with grain size smaller in the constant bed volume will cause internal and external pores greater until total of pores volume in the bed or usually expressed by effective porosity will greater. Effective porosity result



Fig 6. Effect of minerals grain size on effective porosity

Table 1. Influence of Zeofile-to-quartz weight ratio on physical characteristics									
Composition of zeolite/quartz, wt.%	Physical characteristics						Sum of		
	Рь g/cm <sup>3</sup>	Рь score	ε	ຣ score	K cm/dt	K score	score		
100/0	0.649	1	0.737	6	4.064. 10 <sup>-3</sup>	1	8		
80/20	0.795	2	0.705	5	3.886. 10 <sup>-3</sup>	2	9		
60/40	0.832	З	0.678	4	3.345. 10-4	6	13		
40/60	0.893	4	0.657	З	7.175. 10 <sup>.4</sup>	5	11		
20/80	1.005	5	0.662	2	9.203. 10 <del>-4</del>	4	12		
0/100	1.258	6	0.636	1	9.845. 10 <sup>-4</sup>	3	10		

Influence of zeolite-to-quartz weight ratio on physical characteristics



Permeability, cm/second

Fig 7. Effect of minerals grain size on permeability

of mineral for grain size has been with theory expressed by Ven Te Chow [10].

Fig. 6 and 7 showed that zeolite with grain size of 0.196 mm gave the bigest effective porosity and the smallest permeability, whereas quartz with grain size of 0.116 mm gave the bigest effective porosity and the smallest permeability. According to this result, then zeolite with grain size of 0.196 mm and guartz with grain size of 0.116 mm are used as the mixture of local mineral to determine physical characteristics in the backfill material mixture of zeolite-quartz.

The effect of zeolite-to-quartz weight ratio on physical characteristics shown in Table 1. Quartz with grain size of 0.116 mm used is intended to fill pores between zeolite particles in the bed of adsorbent. This addition will improve physical characteristics of the zeolite e.g. bulk density and porosity of the backfill material to be bigger, permeability of the backfill material to be smaller because pores between zeolite particles filled with guartz particles having smallest grain size.

The biggest bulk density can increase the mechanical strength in holding canister of the radioactive waste in the repository such as certained by Atomic Energy Canada Limited [1].

The smaller grain size of adsorbent in the experimental column will increase surface area and effective porosity of adsorbent particles. The bigger effective porosity will increase total of liquid filled internal pores of adsorbent bulk. Possibility of contact between

**Table 2.** Distribution coefficient (K<sub>d</sub>) and dispersivity  $(\alpha)$  of uranium in the backfill material formula

Backfill material formula (wt.% )	Distribution coefficient of uranium, K <sub>d</sub>	Dispersivity of uranium, α (cm)
	(cm <sup>3</sup> /g)	
0.196 mm Zeolite	36.250	0.818
(100 wt. %)		
0.116 mm Quartz	5.661	0.847
(100 wt. %)		
0.196 mm Zeolite / 0.116	18.071	0.759
mm Quartz (60 / 40 wt. %)		

between adsorbent grains with solution contain of uranium is bigger so that adsorption of uranium by zeolite become bigger. The smaller permeability of zeolite-quartz mixture, the greater retardation of solution stream and the more extensive contact time of solution with adsorbent.

When showed from result of physical characteristics on various of zeolite-quartz mixture composition, then zeolite-quartz composition by zeolite-to-quartz weight ratio of 60/40 wt. % is the best with score total of physical characteristics of 13. Base on this case, then zeolite-quartz composition with zeolite-to-quartz weight ratio of 60/40 wt. % can be proposed as candidate of raw material of backfill material.

Base on the result of analysis of UO22+ concentration in influent and effluent of artificial waste solution of uranium, the distribution coefficient can be computed, representing the amount of  $UO_2^{2+}$  in the solution adsorbed by backfill material, dispersivity i.e. represents  $UO_2^{2^+}$  dispersion in the backfill material caused by the difference of  $UO_2^{2^+}$  in the solution and in the particles of the backfill material, and convection that causes the flowing of fluid. The effect of backfill material formula on the uranium sorption characteristic was represented by uranium distribution coefficient  $(K_d)$ and uranium dispersion characteristic was represented by dispersivity ( $\alpha$ ) shown in Table 2.

The experiment result shown in Table 2 can be explained that uranium distribution coefficient (K<sub>d</sub>) in

the zeolite mineral of 36.25 cm<sup>3</sup>/g. This case is caused by the fact that zeolite used was natural zeolite. Natural zeolite contains impurities such as K, Na, Ca, Ba, Mg, and Fe cations that fills interstice of micelle of zeolite aluminosilicateste crystal to maintain the neutrality of the mineral charge. So the potential of UO2<sup>2+</sup> change for the impurities cations in the natural zeolite will not be that good as that of active zeolite. This can be understood, because in the active acid used for an active agent,  $UO_2^{2^+}$  cation only exchanged for H<sup>+</sup> cation of the Hzeolite or Na<sup>+</sup> cation of the Na-zeolite if NaCl used. Quartz gives uranium distribution coefficient (K<sub>d</sub>) of 5.661 cm<sup>3</sup>/g, this result is smaller than  $K_d$  of natural zeolite. This can be understood, because sorption of solute in the zeolite was caused by physical and chemical sorption, whereas in the guartz was due to physical sorption only. Physical sorption of uranium molecule will be filtrated in the cavity of among particles or to stick on the particles surface. Chemical sorption can occur because cations of K, Na, Ca, and Ba in the zeolite on outer framework structure of aluminosilicates will be exchanged by  $UO_2^{2^+}$  cation. In addition, chemical sorption can occur because  $UO_2^{2^+}$  cation is pulled by negative charge on the aluminosilicates crystal lattice. This was caused by the isomorphous replacement of  $AI^{3+}$  for  $Si^{4+}$  gives rise to a negative charge on the lattice. Such a replacement is common to all aluminosilicates, clays included, and the net negative charge must be compensated by cations to preserve the electroneutrality [14].

The addition of quartz to zeolite will decrease the adsorption potential of uranium by solid particles as represented by uranium distribution coefficient. The decrease of uranium distribution coefficient was caused by part of zeolite exchanged by quartz with the adsorption potential smaller than that of natural zeolite.

The addition of quartz with grain size of 0.116 mm having permeability smaller than that of zeolite with grain size of 0.196 mm in the backfill material formula of zeolite-quartz will decrease linearity with velocity of artificial waste liquid leached ( $V_w$ ). The smaller linear velocity causes longer contact time of  $UO_2^{2^+}$ , so that potency of zeolite particles to adsorb  $UO_2^{2^+}$  is maximum. This is may be caused the mass transfer of  $UO_2^{2^+}$  from high concentration to low concentration. The mechanism of  $UO_2^{2^+}$  adsorption from the fluid by zeolite particles happened on the surface, edge, and lattice of zeolite crystal. In this case, zeolite functions as molecule filter and ion exchanger.

Backfill material from the mixture of 0.196 mm diameter zeolite and 0.116 mm diameter quartz with composition of zeolite-to-quartz weight ratio of 60/40 wt. % gives the best physical characteristics (bulk density, porosity, permeability), sorption characteristic (distribution coefficient), and dispersion characteristic (dispersivity). At this composition, the two materials (zeolite and quartz) is interrelated / interdependent. Distribution coefficient and porosity of zeolite are higher than that of quartz, but dispersivity and bulk density of zeolite lower than that of quartz. In the backfill material, zeolite functions as an ion exchanger, whereas quartz functions to strengthen in its physical conditions. Zeolite usually has bad physical properties such as low thermal conductivity, low resistance to radiation and react with acid.

On the other hand, quartz in the backfill material has good properties such as long-term chemical and physical stability under temperatures, pressures and condition of groundwater so that it can improve the physical properties of the backfill material due to the following reasons :

- a. SiO<sub>2</sub> content in the quartz higher than that of zeolite so that the physical strength of backfill material will increase and resistance to heat of radiation is occured due to immobilized radioactive waste,
- b. Bulk density of the quartz higher than that of zeolite so that will be improved load-bearing capacity of radioactive waste container,
- c. Grains of the quartz can increase the dissipation potential of high localized stresses by means of distribution the stress concentration at one point to surrounding rock evently if the earth quake conditioned [1].

Backfill material formula from a mixture 0.196 mm zeolite and 0.116 mm quartz with zeolite-to-quartz weight ratio of 60/40 wt. % give physical characteristic represented by permeability (K) of 3.345.10<sup>-4</sup> cm/second and dispersion characteristic represented by uranium dispersivity of 0.759. This case can be explained that space of uranium migration in the backfill material formula consists of zeolite-quartz mixture is smaller if than that of backfill material consists of the zeolite or quartz only.

The experiment result has been done by Lopez [1] on the backfill material formula from a mixture of 19.5 mm diameter agregate and 6 wt. % clay give permeability (K) of the backfill material of  $5.3.10^{-4}$  cm/second. Whereas LeNeveu [2] has been achieved K of the backfill material from phyllosilicates mineral of  $2.2.10^{-6}$  cm/second.

If K data from this experiment result was compared with K data from experiment result done by Lopez [1] and LeNeveu [2], accordingly the backfill material formula from a mixture of 0.196 mm diameter zeolite and 0.116 mm diameter quartz by zeolite-toquartz weight ratio of 60/40 wt. % can be proposed as candidate of raw material of backfill material.

## CONCLUSION

From the experimental results it can be concluded that a mixture of 0.196 mm diameter zeolite and 0.116 mm diameter quartz with weight ratio of 60/40 wt. % with permeability of  $3.345.10^{-4}$  cm/second and uranium dispersivity of 0.759 cm can be proposed as candidate of raw material of backfill material in the radioactive waste repository.

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