

Ion exchange capacities and surface areas of some major components and common fracture filling materials of igneous rocks

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This report concerns a study which was conducted for SKBF/KBS. The conclusions and viewpoints presented in the report are those of the author(s) and do not necessarily coincide with those of the client.

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ION EXCHANGE CAPACITIES AND SURFACE AREAS OF SOME MAJOR COMPONENTS AND COMMON FRACTURE FILLING MATERIALS OF IGNEOUS ROCKS

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## SUMMARY

Surface/mass ratios have been determined by the ethylene glycol method and the BET-method, and an- and cation exchange capacities vs pH have been measured by an isotopic dilution technique for some 30-35 pure minerals. These minerals, which are representative of major components and common accessory minerals, including fracture minerals, of crystalline rocks, have also been used in radionuclide sorption studies within the Swedish waste program.

Generally, the anion exchange capacities are low (< 1 meq/kg) for all the minerals. The cation exchange capacities, which increase with pH, are  $\leq 1$  meq/kg for the non-silicates with few exceptions, 1-10 meq/kg for most of the silicates except the phyllosilicates and ranging from 10 up to 800 meq/kg for clay minerals (e.g. montmorillonite) and some of the zeolites (e.g. stilbite).

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## 1. INTRODUCTION

High-level radioactive wastes and spent fuel will be placed in underground repositories in crystalline rock according to the present Swedish concept for waste disposal (1). The most important pathways from the repository to the biosphere for the radionuclides that eventually will be released from the waste canisters would be the open water-carrying fractures of the rock. The retention of these radionuclides, due to interactions with water-exposed mineral surfaces, would constitute a major geologic barrier for many of the elements in question.

The minerals in the fractures, both open water-carrying and healed, are usually quite different from the bulk of the rock. For igneous rocks like granite the major rock-forming minerals would be quartz, feldspars, micas and amphiboles, and also pyroxenes and olivine in basic rocks like basalt (2).

Essentially three categories of minerals are found in fractures:

- weathering and alteration products of the major rock components (clay minerals etc.);
- o metamorphic and hydrothermal products;
- o precipitates from aqueous solutions at low temperatures.

In order to allow a quantitative description of the radionuclide retardation it is essential that the chemical properties of the pathways in the bedrock/groundwater system are characterized in detail.

The following projects, related in various ways to the mineralogy of water-carrying factures and radionuclide migration in these pathways, are in progress within the Swedish waste program:

- o Characterization of fracture minerals (3,4);
- Sorption of radionuclides on fracture minerals and rock-forming minerals (5-7);
- Diffusion of radionuclides through fracture coatings into the underlying bulk rock (8-9);
- o Migration of radionuclides in natural fractures (10-13).

The surface/mass ratio and exchange capacities (cations and anions), for common fracture minerals as well as for some other accessory minerals and major components of igneous rocks, are given in this report. These data have been determined to serve as a basis for further discussions and analyses of the observed radionuclide sorption and migration behaviour in fracture systems.

## 2. SELECTION OF MINERALS

Some minerals representative of crystalline rocks were selected for surface/mass ratios and capacity measurements, as well as for batch-wise sorption studies. These minerals include

- o major rock-forming minerals,
- o identified fracture minerals from drilling cores,
- o other common accessory minerals.
- Included were also some common minerals
- o containing small amounts of actinides or lanthanides,
- o representative of all the six major silicate classes.

The selected minerals are listed in Table 1.

Pure well-characterized mineral fractions were crushed and sieved. The 0.044-0.063 mm fraction was washed and gently dried at 105°C (except for some of the clay minerals, see below) and used in the surface and capacity measurements.

#### 3. EXPERIMENTAL

## 3.1. Surface areas

Surface/mass ratios were measured by the ethylene glycol method (E.G.)  $(\underline{14-16})$ . A dried sample (200-1000 mg) was wetted by ethylene glycol followed by equilibration in an evacuated desiccator containing anhydrous calcium chloride-ethylene glycol solvate until the weight was constant. The remaining ethylene glycol layer on the mineral grains has a uniform thickness, and the total surface can be calculated from the weight difference between the untreated and the ethylene exposed sample. Relative errors are large; a minimum of 10-20% for values below 5 m<sup>2</sup>/g and possibly decreasing down to 5-10% at higher values.

Surface areas were also determined by the BET-method (17) for some of the minerals. The relative error is at least 10-15% in these measurements.

#### 3.2. Exchange capacities

Anion and cation exchange capacities (AEC and CEC, meq/kg) were determined

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Class		Name <sup>a</sup>	Remark	
1.	Sulfides	Galena		
		Chalaopurita		
		Burrhotito		
		Cinncher		
		Durito	F	
2	Ovidos	Corundum	r	
۷.	Hydrovidee	Hematite		
	nyuroxides	Pyrolugite		
		Magnetite		
		Limonite/Goethite		
		Cibbsite		
3	Halides	Fluorite		
5.	Carbonates	Calcite	F	
	Sulfates	Dolomite	F	
	Phosphates	Gypsum/Anhydrite	- 7	
	1	Apatite		
4.	Silicates	1		
	a. nesosilicates	Olivine	М	
		Garnet/Almandine		
		Zircon		
		Sphene		
	b. sorosilicates	Epidote	F	
	c. cyclosilicates	Beryl		
	c. inosilicates	Augite		
		Hornblende	М	
		Attapulgite		
	d. phyllosilicates	Serpentine		
		Kaolinite	F	
		Halloysite		
		Montmorillonite	F	
		Palygorskite	F	
		Muscovite	М	
		Biotite	M,F	
		Chlorite	F	
		Prehnite	F	
	e. tectosilicates	Quartz	M,F	
		Microcline	М	
		Albite	М	
		Anorthite/Bytownite	М	
		Analcime	F	
		Stilbite	F	
		Laumontite	F	

Table 1 Minerals selected for studies of anion and cation exchanges capacitites (AEC and CEC) and determinations of surface areas.

<sup>a</sup> See description and composition e.g. in ref. <u>2</u>. Detailed information concerning the particular mineral samples investigated in this study (origin, chemical composition, X-ray diffraction data etc.) are available but not included in this report.
 <sup>b</sup> Excitation function of superclaims reach (2, 4)

F = identifed in fractures of crystalline rock (3, 4).

M = Major component in igneous rocks.

for the minerals as a function of pH. An isotopic dilution technique was employed (<u>18</u>), using <sup>82</sup>Br (35h) and <sup>22</sup>Na (2.6y) as tracers in the saturation of the mineral samples with Br<sup>-</sup> and Na<sup>+</sup>, respectively. A minimum of 3-4 samples were exposed to a spiked fairly concentrated NaBr-solution (0.5 M or higher) of known specific activity and of various pH (5-10). The minerals were saturated with respect to Na<sup>+</sup> and Br<sup>-</sup> (total contact time of 96h), and pH was measured. The aqueous phase was removed by centrifugation and the solid was thoroughly washed with methanol (5 times) to remove any remaining NaBr-solution. The total amount of <sup>22</sup>Na and <sup>82</sup>Br remaining on the solid was determined, and the amount of only <sup>22</sup>Na after 6 weeks of storage, when the <sup>82</sup>Br had decayed almost completely. The amounts of exchangeable Na<sup>+</sup> and Br<sup>-</sup> were calculated from the known specific activites.

## 4. RESULTS AND DISCUSSION

Calculated surface/mass ratios are given in Table 2. The values obtained by the E.G.-method would possibly be representative of the surfaces available for interactions with species in aqueous phases in contact with the solids. The E.G.-surfaces are generally larger than the BET-surfaces for the same solid material, Fig. 1. The correlation between E.G.- and BET-surface data is, however, acceptable, considering the relative errors of these measurements.

The measured exchange capacities (AEC and CEC) are given in Fig. 2-10 and summrized in Table 3. The anion exchange capacity is generally low both on non-silicates and silicates, rarely above 1 meq/kg. Exceptions are limonite and pyrolusite among the oxides/hydroxides (surface effects; small particles) and some of the silicates like olivine, zircon, attapulgite, muscovite, bytownite and laumontite which have values around or slightly above 1 meq/kg. High values (above 10 meq/kg) were found for some of the zeolites (analcime, and particularly stilbite) as well as for palygorskite, and occasionally for limonite (not given in Table 3). The AEC is slightly decreasing with increasing pH.

The cation exchange capacity is generally low for the halides, sulfates and phosphates among the non-silicates (< 1 meq/kg), and usually significantly higher, but still low (around 1 meq/kg) for the oxides. Exceptions are limonite and pyrolusite (surface effects).

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Mineral	Surface/mass ratio, m <sup>2</sup> /g			
minerar	Ethylene glycol (E.	G.) BET	BET	
Chalcopyrite	7.2	1.0		
Cinnabar	7.7	1.9		
Pyrite	7.0			
Hematite	1.8	0.9		
Magnetite	2.1	1.5		
Corundum	2.1	0.5		
Limonite	80	13		
Gibbsite	13			
Fluorite	1.6			
Calcite	2.9	0.6		
Dolomite	4.0			
Anhydrite	3.8			
Apatite	2.8	0.6		
Olivine	5.0	2.0		
Zircon	4.1			
Garnet/Almandine	2.1			
Sphene	3.3			
Epidote	4.2			
Augite	6.3			
Hornblende	4.6	4.3		
Attapulgite <sup>a</sup>	340	190		
Serpentine	61	15.7		
Kaolinite	27	11		
Halloysite	93	32.0		
Montmorillonite	800			
Muscovite	11	7.2		
Biotite	14	7.0		
Chlorite	24			
Quartz	2.8	0.3		
Microcline	4.2	1.1		
Albite	2.9			
Bytownite/Anorthite	3.1			

# Table 2 Surface/mass ratios.

a Non-sieved material



<u>Figure 1</u> Correlation between surface/mass ratios (A) as obtained by the BET-method and the ethylene glycol method (E.G.). O non-silicates, • silicates.

Most silicates except the phyllosilicates and the zeolites among the tectosilicates have CEC-values in the range 1-10 meq/kg. For the phyllosilicates and zeolites values from 20-50 meq/kg (e.g. attapulgite, kaolinite, halloysite, muscovite, biotite, chlorite, laumontite) are usually observed, however, with the exceptions of some extreme high-capacity minerals with capacities of several hundred meq/kg (montmorillonite, analcime, stilbite).

The cation exchange capacity shows a pronounced pH-dependence (increasing CEC with pH) which is reasonable considering the charge vs pH of the mineral surfaces.

Thus, a degradation and weathering of e.g. feldspars into clay minerals can lead to an enhanced CEC by up to more than two orders of magnitude. It is obvious, that the composition of fracture minerals must have a large influence on the concentrations of cations in the water in contact with the clays ( $H^+$ ,  $Na^+$ ,  $K^+$ ,  $Mg^{2+}$ ,  $Ca^{2+}$ ) through ion exchange equilibria. This could be one reason for the close similarity in concentrations of these elements in deep groundwaters from a variety of geologic systems.



Figure 2 Exchange capacities for sulfides.

a. AEC; b. CEC.

■ galena, ◇ sphalerite, ● chalcopyrite,
□ pyrrhotite, ○ pyrite.



Figure 3 Exchange capacities for oxides and hydroxides. a. AEC; b. CEC.





Figure 4 Exchange capacities for halides, carbonates, sulfates and phosphates. a. AEC; b. CEC.

○ fluorite, ■ calcite, ● dolomite,
□ anhydrite, ◇ apatite.



Figure 5 Exchange capacities for neso- and sorosilocates. a. AEC; b. CEC. ○ olivine, ● almandine, ■ zircon, □ sphene, ◇ epidote, ● prehnite (phyllosilicate).



Figure 6 Exchange capacities for cyclo- and inosilicates.
 a. AEC; b. CEC.
 O beryl, ● augite, □ hornblende,

serpentine (phyllosilicate).



Figure 7 Exchange capacities for phyllosilicates.
a. AEC; b. CEC.
O kaolinite, ● halloysite, ■ montmorillonite,
Ø palygorskite, □ attapulgite (inosilicate).



Figure 8 Exchange capacities for phyllosilicates.
a. AEC; b. CEC.
○ montmorillonite, □ muscovite, ● biotite, ■ chlorite.





Figure 10 Exchange capacities for tectosilicates.
 a. AEC; b. CEC.
 □ analcime, ● stilbite, ○ laumontite,

♦ palygorskite (phyllosilicate)

	AEC, meq/kg		CEC, meq/kg		
	pH 5	pH 8	рН 5	pH 8	
Galena	1.0	1.0	0.9	1.1	
Sphalerite		0.5	1.3	1.4	
Chalcopyrite	0.7	0.3	1.0	2.6	
Pyrrhotite	0.9	0.4	0.8	1.5	
Pyrite	0.2	0.2	1.3	1.5	
Corundum	0.4	0.3	0.2	0.4	
Hematite	0.3	0.4	0.2	0.5	
Pyrolusite	2.0	1.5	5	8	
Magnetite	0.8	0.5	0.2	0.4	
Limonite	3.5	0.7	1.0	6.3	
Fluorite	0.1	0.1	0.2	0.2	
Calcite		0.1		0.2	
Dolomite		0.1		0.2	
Anhydrite	0.1	0.1	0.5	0.8	
Apatite	0.1	0.1	0.4	0.5	
Olivine	2.3	1.7	0.8	1.8	
Zircon	2.3	0.6	1.5	3.5	
Epidote	0.7	0.4	2.8	6.0	
Beryl	2.5	1.8	0.8	4.5	
Augite	0.2	0.2	4.0	8.0	
Hornblende	0.2	0.1	1.8	2.5	
Attapulgite	1.5	0.5	45	85	
Serpentine		6.8	0.4	1.0	
Kaolinite	0.8	0.6	9.0	28	
Halloysite	2.5	1.5	32	45	
Montmorillonite		0.7	700	800	
Palygorskite		10		80	
Muscovite	1.0	0.6		52	
Biotite	0.4	0.3	6	17	
Chlorite	0.3	0.3	50	50	
Prehnite		0.3		25	
Quartz	0.1	0.1	0.1	0.2	
Microcline	0.2	0.1	1.8	3.7	
Albite	0.2	0.1	1.8	3.7	
Anorthite`	3	0.5	2.2	5	
Bytownite	0.3	0.2	6	9	
Analcime	12	12		180	
Stilbite	30	45	630	640	
Laumontite	5	3	35	35	

<u>Table 3</u>	Exchange capaci	ties	at	pH5	and	pH8
	(Extrapolations	from	n F:	ig.	2-10)	)

For many of the silicates there is a significant correlation between CEC and the surface/mass ratio, Fig. 11, and also to some extent for the non-silicates. Also there are significant correlations between radionuclid sorption behaviour and surface/mass ratio and/or exchange capacities, as discussed in detail elsewhere (5-7). This relations can briefly be summarized as follows:

- o The sorption is generally increasing with increasing available cation exchange capacity for non-hydrolyzed spherical cations like  $Cs^+$  and  $Sr^{2+}$ ; to some extent also for  $Am^{3+}$  (at low pH), but to a less extent for species like  $NpO_2^{++}$ .
- o There is only a qualitative correlation (or no correlation at all) between sorption and exchange capacity for hydrolysed or complexed ions (e.g. Am(III) at intermediate pH; U(VI) at intermediate pH in the presence of carbonate).
- o There is usually some minor correlation between sorption and surface/ mass ratio for hydrolysed cations and a more significant correlation for anionic species (e.g. I<sup>-</sup>, ref. <u>19</u>).



Figure 11 Correlation between cation exchange capacity (CEC) and surface/ mass ratio (A).

○ non-silicates, ● silicates.

The presence of weathering products with high CEC in water-filled fractures will have a large impact on the Na-K-Mg-Ca-relations as well as on pH in deep groundwaters (20). Cation exchange reactions would be expected to be much faster than the release of cations due to the progressing weathering processes. These reactions would be rate-controlled by the diffusion of reacting species ( $H^+$ ) and reaction products through a solid layer, although porous and water saturated.

The degree of weathering, crystallinity, particle size distribution, porosity etc. of minerals from various sources vary considerably. The values in Table 2 and 3 are representative of the particular minerals selected in this study and for further sorption measurements reported elsewhere (5-7). Substantially different surface/mass ratios could be obtained for minerals from other sources or by the use of other experimental procedures or size fractions. The presence of small amounts of weathering products of high CEC (e.g. clay minerals) in minerals of low CEC (e.g. feldspars) could give apparent CEC-values that could be more than one order of magnitude higher than the values given in Table 3 in certain systems (micas, feldspars).

## 5. CONCLUSIONS

Some general conclusions are:

- o The anion exchange capacity is low for most of the studied mineral systems and decreasing with pH. Values above 1 meq/kg were only obtained for limonite (surface effect) and for some of the zeolites.
- o The cation is low for all non-silicates and only occasionally above l meq/kg, e.g. for the oxides and sulfides. Slightly higher values are obtained for most of the silicates, and extremely high values are obtained for clayish minerals as well as zeolites (e.g. up to 800 meq/kg) for montmorillonite).
- The weathering and decomposition of rock-forming minerals generally gives products with a strongly increased CEC.
- o Exchange capacities, AEC (for anions like I) and CEC (for cations) have a large and significant influence on the sorption of particularly non-hydrolyzed.

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