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GEOCHEMICAL STUDIES ON THE OCCURRENCE OF THE FLUORIDE-RICH GROUNDWATER IN MIZUNAMI AREA, JAPAN

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ABSTRACT

Fluoride-rich groundwater exceeding the Japanese environmental standard level (0.8 mg/l) was observed in the groundwater of Mizunami area, Japan. Previous research in Mizunami area suggested that the occurrence of fluoride in the groundwater is mainly due to the interaction between fluoride-rich minerals in granitic rock and groundwater. In this study the existence of fluoride in rock forming minerals was investigated by performing mineralogical analysis as well as water-rock interaction test. The fluoride concentration and pH value are suddenly increased at the beginning of the reaction for most of the granitic specimens. The increases of fluoride concentrations are much different among granitic samples, and it implies the spatial change on the distribution of fluoride-rich minerals in granite. X-ray diffraction analysis for rock powder before and after water-rock interaction test showed the existence of fluorite mineral in relatively small amount as comparing with other minerals. Statistical analysis for the results showed that the fluoride ion correlated positively with Ca^{2+} , Na^+ , Si^+ and Al^{3+} ions ion; however, Cl^- have a negative correlation with fluoride ion.

Keywords: Mizunami area, fluoride, granitic rock, water-rock interaction and mineralogical analysis

1. INTRODUCTION

The Japan Atomic Energy Agency (JAEA) has been constructing a large underground research facility named as the Mizunami Underground Research Laboratory (MIU) at the Mizunami city, Gifu prefecture, central Japan. During the construction of two 1,000.0 m shafts at the MIU site, much amount of groundwater with a high fluoride concentration discharged into the shaft. The fluoride ion concentrations of the groundwater significantly exceeded the Japanese standard allowance of 0.8 mg/l (JNC, 2000; Arthur et al., 2006), so that a special treatment facility was constructed to decrease the concentration of fluoride in the discharged water. Iwatsuki *et al.* (2005) studied the hydrochemistry of the groundwater around the MIU-site. They clearly pointed out the existence of high concentrations of fluoride ions in the groundwater. Abdelgawad et al. (2007) performed a water-rock interaction test to study the occurrence of fluoride-rich groundwater in Mizunami area. It was suggested that the high fluoride concentrations are mainly due to the interaction between fluoride-rich minerals in granitic rock and groundwater. In this study, we extended the water-rock interaction test

from the previous research by increasing the numbers of specimens and the reaction time also mineralogical analysis was performed to investigate the origin of fluoride-rich groundwater in Mizunami area.

2. DESCRIPTION OF THE STUDY AREA

The Mizunami Underground Research Laboratory project (MIU) began in 2002 to investigate crystalline rock in Mizunami city, Gifu, central Japan. Figure 1 shows the location of the MIU-site and the boreholes drilled in and around the site for the hydrogeological investigation. The main goals of the MIU project are to establish techniques for investigation the deep geological environment and acquisition of hydrochemical data for use in deep underground research (JNC, 2002). The MIU essentially consists of two 1,000 m shafts (the Main and the Ventilation Shafts) and two principal research levels. Four shallow boreholes (MSB-1, 2, 3 and 4) and one deep inclined borehole (MIZ-1) have been constructed at the surrounding area of the MIU for monitoring the effect of shaft excavation on groundwater flow and the groundwater chemistry, Figure 1.

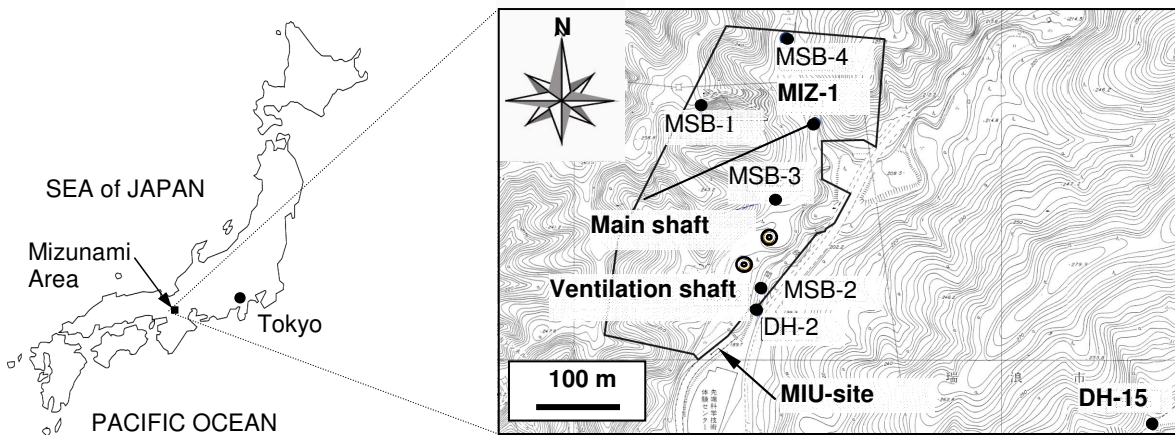


Figure 1 location of MIU-site

In Tono area, Tertiary sedimentary rocks called Mizunami Group (several hundred meters thickness) unconformably overlie the Cretaceous crystalline basement (the Toki granite). The Mizunami Group at the MIU site is divided into Akeyo Formation, Hongo Formation and Toki Lignite-bearing Formation in descending order below ground surface. The top of the Toki granite varies in elevation from 50.0 to 150.0 masl (meters above sea level) at the MIU construction site. A thin layer in the upper part of the Toki granite is highly weathered. The thickness of the weathered layer observed along deep borehole MIZ-1 is around 10 m, but this thickness varies from place to place. The main body of the Toki granite is classified as medium-to-coarse grained. The Toki granite is divided into two structural domains based on the intensity of fracturing: the Upper Highly Fractured Domain (UHFD) and the Lower Sparsely Fractured Domain (LSFD). Many low angle fractures (jointed zone) have been identified in the UHFD and called as Low Angle Fractured Zone (LAFZ), as shown in Figure 2(a). The most dominant oxides in sedimentary and granitic rock of MIU-site are showing in Table 1.

Iwatsuki *et al.* (2005) constructed a hydrochemical conceptual model in the area of MIU-site and concluded that the groundwater mainly divided into two types; the Na-(Ca)-HCO₃ type and the Na-(Ca)-Cl type. The former type is mainly distributing in the Akeyo and Hongo Formations. The later type is evolved in the Toki lignite bearing Formation and Toki granite and it can be subdivided into two types. The groundwater shallower than

approximately -400 masl is considered as low Na-(Ca)-Cl water-type. On the other hand, the deep part of granite is dominated by a high Na-(Ca)-Cl water-type, as schematically evidenced in Figure 2 (b). The possible salinity source in the MIU area may be occurred when the region inundated with sea water during marine transgression in the Miocene. The infiltrating rainwater has been flushing the old marine water contained in the rock mass. Rainwater is mainly infiltrating at higher altitude area (recharge area) and flowing towards the lower area (discharged area). Old marine water is more flushed in the recharge area developing at the north of the site.

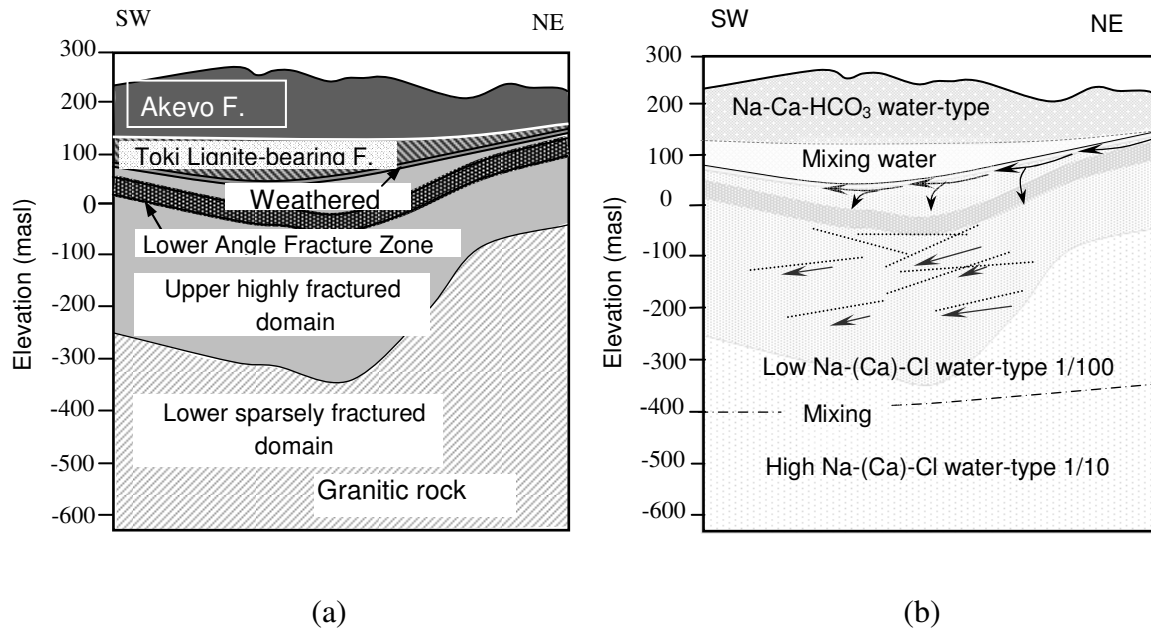


Figure 2 a) Schematic geological cross-section at MIU site. b) Conceptual hydrogeochemical model in and around MIU construction site. Arrows refer to groundwater flow paths. Rewrite after Iwatsuki et al. (2005).

Table 1 Concentrations of oxides (wt%) in sedimentary and granitic rock of Tono area*.

B.h. No.	Geology ^a	Depth m	SiO ₂ %	Al ₂ O ₃ %	Fe ₂ O ₃ %	CaO %	MgO %	Na ₂ O %	K ₂ O %	MnO %	P ₂ O ₅ %
MSB-1	A.	27.3	46.15	10.76	3.16	13.15	0.75	1.66	1.37	0.19	0.10
MSB-1	A.	85.8	55.29	14.30	7.44	5.99	4.80	2.65	1.01	0.18	0.13
MSB-1	T.	173.3	48.41	20.22	6.01	2.73	1.36	1.99	1.16	0.06	0.04
MSB-2	A.	35.6	82.76	5.46	2.47	1.23	0.56	0.91	0.58	0.03	0.04
MSB-2	T.	171.8	73.01	10.94	2.19	2.31	0.26	1.90	3.85	0.07	0.04
MSB-3	A.	20.9	50.30	7.53	3.72	15.93	1.50	1.35	0.69	0.45	0.21
MSB-3	T.	166.6	61.30	10.29	2.02	10.31	0.47	2.42	3.17	0.18	0.28
MSB-4	A.	34.3	60.83	10.41	2.02	10.60	0.41	2.39	3.15	0.18	0.28
MSB-4	T.	81.7	70.20	12.13	2.45	2.16	1.20	2.48	4.12	0.04	0.13
MIZ-1	G.	121.1	70.8	13.0	3.0	1.22	0.13	3.2	4.3	0.14	0.2
MIZ-1	G.	295.0	69.58	13.17	3.12	1.26	0.22	3.31	4.34	0.16	0.19
MIZ-1	G.	369.1	70.58	13.15	2.7	1.11	0.05	3.34	4.58	0.22	0.11
MIZ-1	G.	490.1	69.1	15.0	2.58	1.37	0.13	3.45	5.19	0.16	0.17
MIZ-1	G.	608.0	67.9	15.0	3.23	1.3	0.17	3.34	5.7	0.18	0.17
MIZ-1	G.	706.0	69.84	13.07	2.78	1.41	0.18	3.53	4.56	0.13	0.17
MIZ-1	G.	984.12	74.75	10.27	2.52	0.74	0.04	3.21	3.98	0.22	0.08
MIZ-1	G.	1180.1	71.0	12.97	2.54	0.8	0.14	2.79	5.25	0.11	0.17
MIZ-1	G.	1238.0	70.89	12.13	2.91	1.0	0.14	3.04	4.67	0.19	0.15
MIZ-1	G.	1298.12	73.0	12.0	2.79	1.06	0.14	3.6	3.82	0.15	0.22

* Uozumi et al., (2005).

^a A: Akeo Formation, T. Toki lignite-bearing Formation. And G. Toki granite

3. COLLECTING AND PREPARING OF ROCK SPECIMENS

This study is focusing mainly in two approaches; mineralogical analysis and water-rock interaction test. At first, the fluoride contents in granitic rock were measured by collecting several granitic specimens from a boring core of a deep inclined borehole drilled at Mizunami Underground Research laboratory construction site (MIU-site) named MIZ-1 borehole. Nine granitic rock specimens were collected from different depths of the core MIZ-1 borehole. These specimens were collected from different depths in the granite basement; the highly weathered zone, slightly altered zone, upper highly fractured domain, lower sparsely fractured domain and extremely altered granite. Table 2 summarizes the depths of the sampling points as well as the characteristics of the rock specimens.

Thin sections of some of these specimens of 30 microns thickness were prepared to investigate the mineral conditions of those granitic specimens and their textures by using optical microscopy. For the water-rock interaction test, the granitic rock specimens were crushed in a ceramic shatter box to make rock powder. The wet sieving technique was used to divide the powder into three sizes (0.30-0.15 mm, 0.15-0.075 mm and smaller than 0.075 mm).

Table 2 Locations and characteristics of rock specimens collected from borehole MIZ-1.

Samples	Depth mabh ^a	Elev. Masl ^b	Geology		
			layer	Structural ^c	Weathering/ Alteration
MIZ-1-G1	110.33	96.26	G	UHFD	highly weathered
MIZ-1-G2	131.10	75.46	G	UHFD	slightly altered
MIZ-1-G3	151.35	55.21	G	UHFD	slightly altered
MIZ-1-G4	222.10	-15.54	G	LAFZ	highly altered
MIZ-1-G5	335.40	-128.71	G	LSFD	unaltered
MIZ-1-G6	546.00	-336.12	G	LSFD	slightly altered
MIZ-1-G7	654.45	-442.20	G	LSFD	highly altered
MIZ-1-G8	801.30	-585.85	G	LSFD	highly altered
MIZ-1-G9	976.20	-756.90	G	LSFD	extremely altered

^a mabh: Meter around borehole.

^b masl: Meter above sea level.

^c UHFD: Upper Highly Fractured Domain, LSFD: Lower Sparsely Fractured Domain and LAFZ: Low Angle Fractured Zone.

4. CHARACTERIZATION OF ROCK IN MIU SITE

4.1 Sedimentary rock

In the area around MIU-site, Miocene sedimentary rocks of Mizunami group unconformably overlie a basement of Cretaceous granitic rock at depths about 100-200 m. The upper part of the Mizunami group (Akeyo F.) are composed of mudstone and silt stone-sandstone alternations with shallow marine facies. The middle and lower parts of the Mizunami group, (Hongo F. and Toki Lignite-bearing F.) is composed of tuff, siltstone-sandstone and conglomerate. Major constituent minerals of sedimentary rocks are quartz, feldspar, plagioclase, amphibole and clay minerals. In the Akeyo formation gypsum and

minor amounts of biotite are present and magnetite and ilmenite are rare (JNC, 2000; Arthur et al., 2006). The mineralogy of Toki lignite-bearing formation is determined by point counting method as follows (Iawatsuki et al., 1995): quartz (0.1%), plagioclase (21.5%), biotite (0.3%), amphibole (0.6%), calcite (0.1%), zeolite (0.4%), clay minerals (76.8%), opaque minerals (0.8%) including pyrite. The dominant mineral is clay mineral such as smectite, which have a relatively high ion exchange capacity [cation exchange capacity of 0.56 meq/mg and exchangeable Na content of 0.12 meq/mg and exchangeable Na content of 0.12 meq/mg].

4.2 Granitic rock

The basement granitic rocks consist of medium - to coarse-grained biotite granite and medium-grained hornblende–biotite porphyry, and are partly intruded by quartz porphyry and aplite dikes. Iwatsuki and Yoshida (1995) have been studied the mineralogy of fracture system in deep granitic rock of the Tono area to understand the chemical evolution of groundwater. Geological studies revealed that fracture system within the granitic rock is usually associated with alteration and it can be classified into intact zones, moderately zones and intensely fractured zones, using the degree of fracturing. In the moderately fracture zone, there is a little alteration, and the filling minerals are mainly chlorite, montmorillonite, and pegmatitic texture. The intensely fracture zones are associated with a strongly altered rock matrix, showing alteration to clay minerals such as kaolinite. The X-ray powder diffraction analyses indicated that the carbonate minerals on the fracture surfaces are mainly calcite (Iwatsuki et al., 2002)

Figure 3 shows the optical microscopy observation of some of the collected specimens. Figure shows that the conditions of minerals suffered by water-rock interaction and/or the past hydrothermal alteration. Figure 3(a) shows section in slightly altered granite (MIZ-1-G4). The major observed minerals are quartz, plagioclase, K-feldspar and biotite. Generally, when the granite is altered, plagioclase and K-feldspar dissolve to form kaolinite and/or montmorillonite, (JNC, 2000). Iron hydroxides such as goethite are observed within minerals such as quartz and plagioclase. The highly and extremely altered granites were represented by core sample MIZ-1-G7 and MIZ-1-G9 (Figure 3(b) and (c)), respectively. The original mineralogical compositions of these hydrothermal altered Toki granites are approximately similar to that of nonaltered granite. It was observed from these figures that the mineral crystals in highly and extremely altered rock were destroyed and chloritized, then the color of these specimens changed into green in color. In addition the highly and extremely altered granite contains small amount of sericite, clays and iron oxides.

Several granite specimens were collected from different depths of the core of the MIZ-1 borehole. Fluoride contents in these granitic specimens were measured (see Figure 4). Results show that the fluoride content varies even among specimens collected from almost the same depth. The range of the fluoride content measured at different depths is 200-1000 mg/kg.

In this study, the granitic powder of three specimens, before and after the water-rock interaction test, were selected to perform X-ray Diffraction analysis; one specimen from upper highly weathered granite (MIZ-1-G1), second from slightly altered granite (MIZ-1-G7) and third one from extremely altered granite (MIZ-1-G9). The results imply the existence of fluorite mineral in relatively small amount as comparing with other minerals. From the analysis some other possible minerals were observed to be sources of fluoride such as; phlogopite (Mg-rich mica $\text{KMg}_3\text{AlSi}_3\text{O}_{10}(\text{F},\text{OH})_2$), Fluorannite (F-rich annite-like biotite $\text{KFe}_3\text{AlSi}_3\text{O}_{10}\text{F}_2$), and polyolithionite ($\text{KLi}_2\text{AlSi}_4\text{O}_{10}\text{F}_2$).

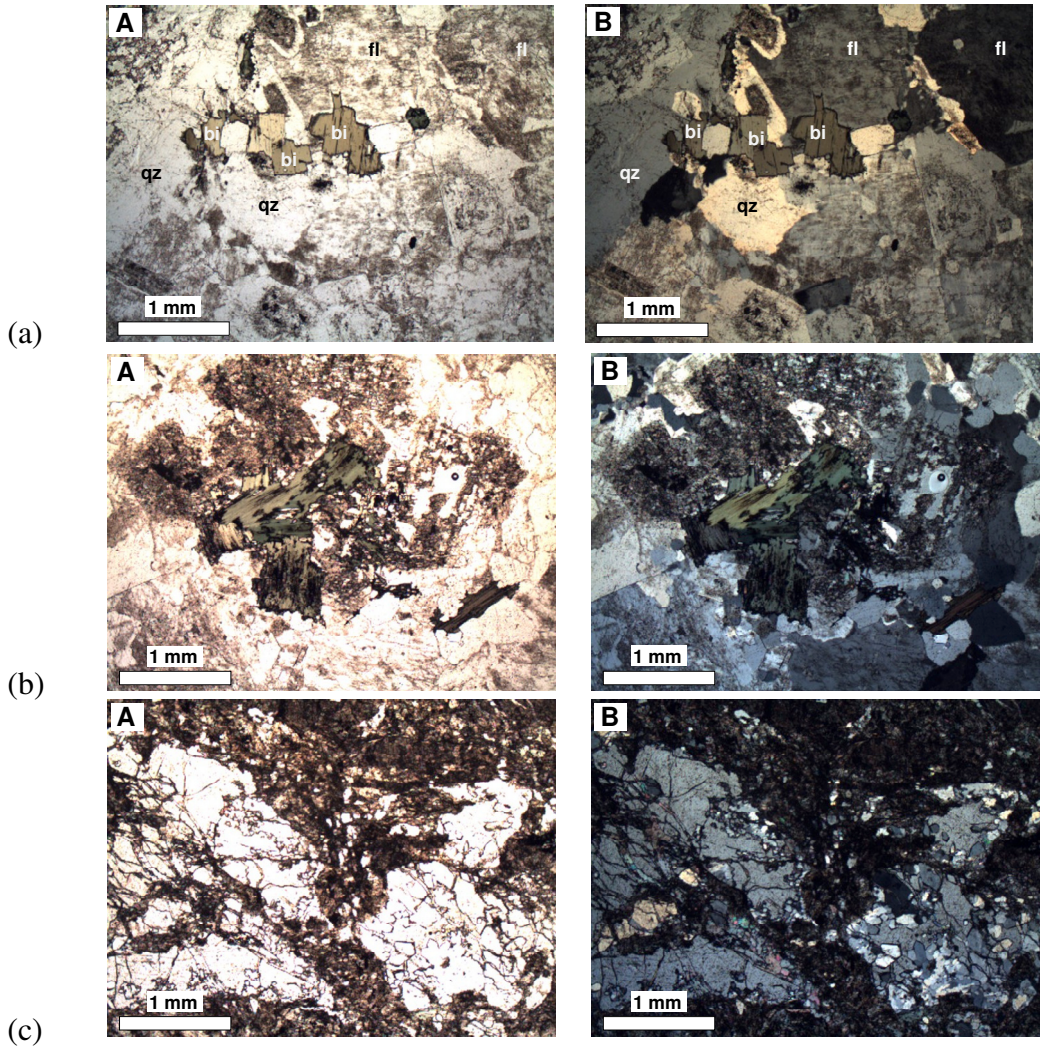


Figure 3. Microscopic observations of granitic specimens.
 (a) Highly weathered (222.10 mbgl) (b) Highly weathered/altered (648.76 mbgl)
 (c) Extremely altered (963.46 mbgl)
 A: open Nicol, B: cross Nicol, qz: Quartz, fl: Feldspar and bi: Biotite.

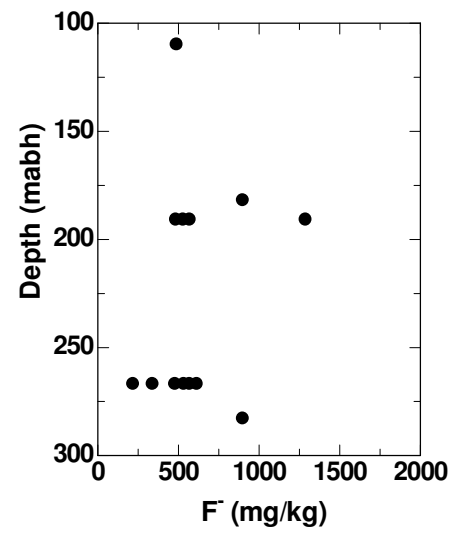


Figure 4. Fluoride content measured from specimens collected from MIZ-1 borehole.

5. WATER ROCK-INTERACTION TEST

A series of batch reaction experiments were performed to investigate the effect of different experimental conditions on the dissolution of fluoride-rich minerals using a shaker table (AS-ONE SRR2). All specimens were reacted with distilled water of pH 6.7 at a constant shaking rate of 180 rpm within tightly capped conical glass flasks under the room conditions (around 20 °C). All batches were designed to have a constant water-rock ratio of 10/1 and the same grain size of powder of 0.15-0.075 mm. For each rock specimen, two batches were conducted in the same conditions. One batch continues for 30 days and other batch continue for 80 days.

For all experiments, the amount of the fluoride ion in the output solutions was roughly measured using the ion selective method (Pack test), and pH was measured using a pH meter (Eutech pH 510). For all experiments continued for 30 days, water samples (30 mm³) were collected from the solutions after 3, 8, 12, 20 and 30 days. For experiments continued for 80 days water samples (30 mm³) were collected from the solutions after 40, 60 and 80 days. Then these water samples were filtered by using filter paper (Whatman No.1) and were then analyzed for cations and anions by using Inductively-Coupled Plasma optical-emission spectrometry (ICP-OES; Varian VISTA-MPX) and Ion Chromatography (IC; Dionex DX-120), respectively. Water samples obtained after 80 days shaking were also used for the analyzing of the HCO₃⁻ concentration.

6. RESULTS AND DISCUSSION

The increase of fluoride concentration is displayed in Figure 5(a). Figure shows that the fluoride ion is enriched for most of the rock specimens with increasing the reaction time even though the pH was almost stable with time. Figure shows the changing on the leaching of fluoride ions with time from different granitic specimens that implies the spatial change on the distribution of fluoride-rich minerals in granite. The highest fluoride concentrations are associated with the extremely altered granitic specimens (MIZ-1-G9). One possible reason for this may be the precipitation of the fluoride ion in the altered zone during the migration of thermal water.

From Figure 5(a and b), it is also clearly found that at the beginning of the reaction of granitic specimens, the sudden increase in F⁻ is associated with sudden increase in pH values up to around 9. After 4-5 days from the start of the experiments the pH decreased and became around 8.0, then it became in the steady condition.

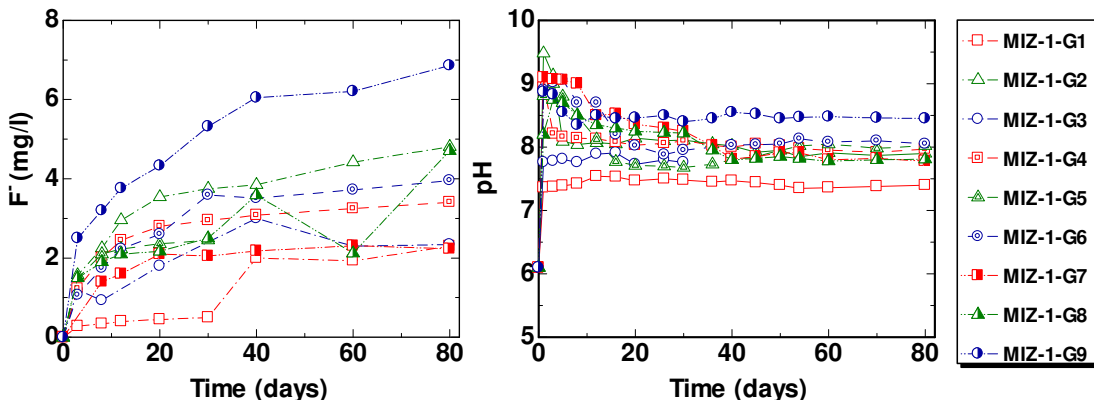


Figure 5. Reaction time versus fluoride concentration and pH for different type of rock..

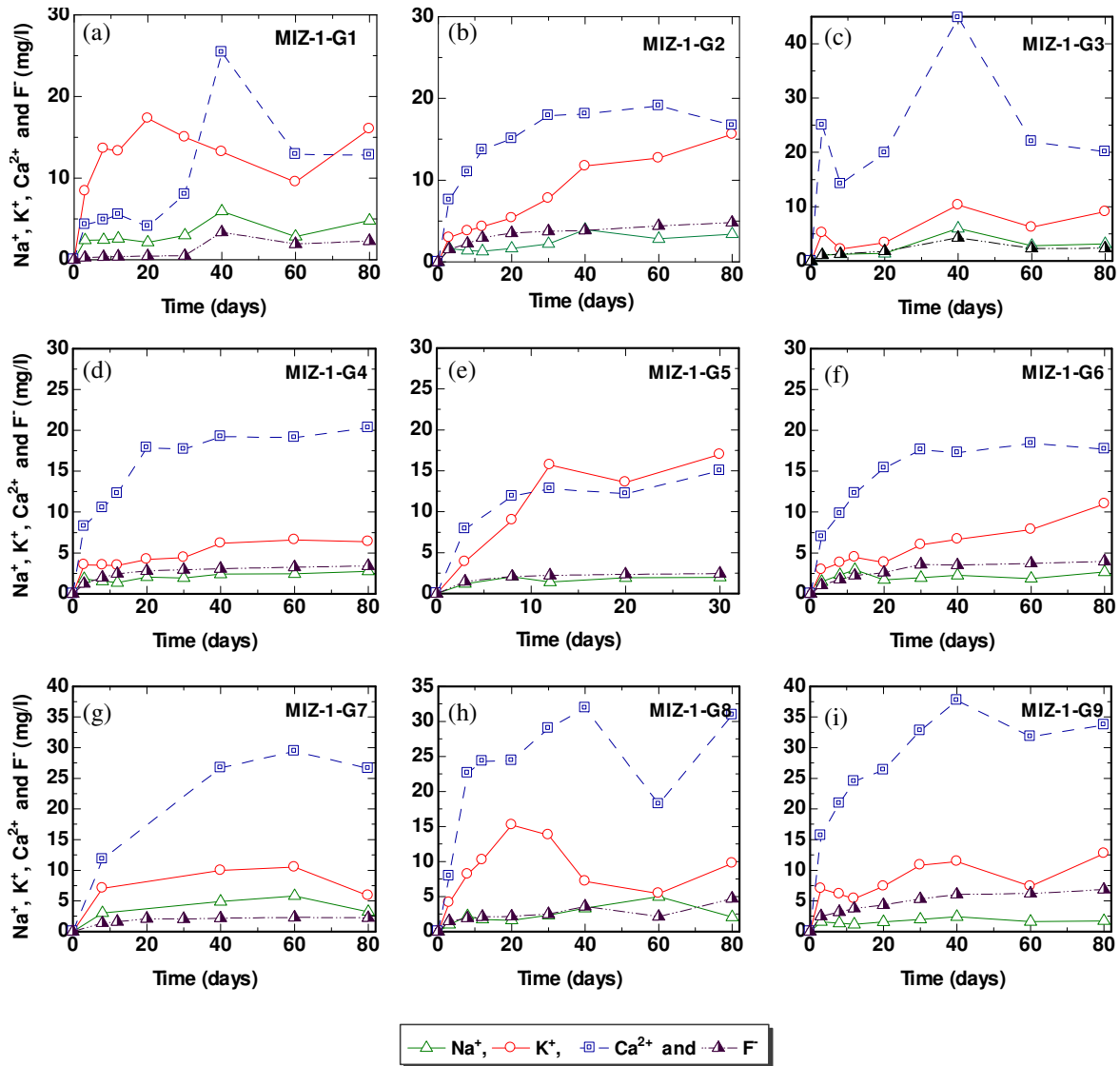


Figure 6. Comparisons between leaching fluoride ions and some selected cations.

Figure 6 shows the leaching of some selected cations (Na⁺, K⁺ and Ca²⁺) in comparison with the leaching of fluoride ions with time. It was clearly observed that among the selected ions, the K⁺ and Ca²⁺ concentrations rapidly increased up to 10 days, as shown in Figure 6. K⁺ concentration increase, may be suggesting the dissolution of biotite (K(Fe, Mg)₃AlSi₃O₁₀(F,OH)₂) and/or K-feldspar within the reaction time. The solubility of Ca⁺ depends on several geochemical processes such as; the dissolution of Ca-bearing minerals and calcite (CaCO₃) precipitation. Iwatsuki et al. (2002) indicated that, the calcite in the Toki granite could have precipitated from marine water in the fracture surfaces. This finding might explain the reason for increasing the calcite concentration leached from deep altered granite (MIZ-1-G8 and MIZ-1-G9) (Figure 6(h and i)) in comparison with other granitic specimens. At the shallow weathered granite (MIZ-1-G1), the hydrolysis of K⁺ ion was faster than that of Ca²⁺ ion, Figure 6(a). This results and X-ray powder diffraction analysis for MIZ-1-G1 specimen before and after shaking suggested that, in the highly weathered granite (MIZ-1-G1), K⁺ might be leached from annite and/or microcline (K-feldspar).

Statistics analysis for the results showed that the fluoride ion correlated positively with Ca²⁺ ion; however, the Na⁺, Si⁺ and Al³⁺ ions have a negative correlation with fluoride ion.

Table 3 shows the correlation coefficient between fluoride and other ions. The positive correlation suggested the continuing dissolution of fluoride leaching minerals and other minerals.

Table 3. Correlation coefficient between fluoride and other ions.

Na ⁺ (ppm)	K ⁺ (ppm)	Ca ²⁺ (ppm)	Mg ²⁺ (ppm)	Si ⁴⁺ (ppm)	Al ³⁺ (ppm)	Fe ^{2+/3+} (ppm)	Cl ⁻ (ppm)	SO ₄ ²⁻ (ppm)	F ⁻ (ppm)
0.33	0.37	0.80	0.76	0.26	0.22	0.20	-0.13	0.54	1.00

7. CONCLUSION

In order to understand the occurrence of fluoride-rich groundwater at the MIU site, a series of experimental studies were performed. The results show that the granite can act as a source of high fluoride concentration due to the increase in the reaction time between the groundwater and the granitic rock. A relatively small amount of fluoride ion is released from highly weathered granite. However fluoride amounts released from extremely altered granite are higher than those of other granitic specimens. Results also showed that coexisting ions on might have an apparent effect on fluoride concentration. Further study for this effect is needed for better understanding the dissolution processes of fluoride leaching form granitic rock.

X-ray diffraction analysis shows that, the change in the fluoride content in the granitic rock due to leaching test for 80 days is invisible and it was difficult to detect the difference on the peak of fluorite mineral before and after water-rock interaction test.

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