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LETTER

Sulfur isotopic characteristics of volcanic products from the September 2014 Mount Ontake eruption, Japan

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Abstract

Components and sulfur isotopic compositions of pyroclastic materials from the 2014 Mt. Ontake eruption were investigated. The volcanic ash samples were found to be composed of altered volcanic fragments, alunite, anhydrite, biotite, cristobalite, gypsum, ilmenite, kaolin minerals, native sulfur, orthopyroxene, plagioclase, potassium feldspar, pyrite, pyrophyllite, quartz, rutile, and smectite, and most of these minerals were likely derived from the acidic alteration zones of Mt. Ontake. The absence of juvenile material in the eruptive products indicates that the eruption was phreatic. The sulfur isotopic compositions of the water-leached sulfate, hydrochloric acid-leached sulfate, acetone-leached native sulfur, and pyrite of the samples indicate that these sulfur species were produced by disproportionation of magmatic SO_2 in the hydrothermal system at temperatures of 270–281 °C. This temperature range is consistent with that inferred from the hydrothermal mineral assemblage (e.g., pyrophyllite and rutile) in the 2014 pyroclastic materials (200–300 °C). Except for the sulfur isotopic compositions of anhydrite, which may have been altered by incorporation of sulfate minerals in a fumarolic area with lower sulfur isotopic values into the underground materials during the 1979 eruption, no significant differences in the mineral assemblages and sulfur isotopic compositions of the pyroclastic materials were identified between the products of the 2014 and 1979 Ontake phreatic eruptions, which suggests geochemical similarities in the underlying hydrothermal systems before the 2014 and 1979 eruptions.

Keywords: Mount Ontake, Phreatic eruption, Sulfur isotopic composition, Hydrothermal minerals

Introduction

Mount Ontake (35°53′34″N, 137°28′49″E; Fig. 1) is the second highest volcano in Japan (elevation: 3067 m). Few phreatic eruptions have been reported around the Jigokudani and Hachotarumi areas in recorded history (Oikawa 2008). On September 27, 2014, this volcano suddenly erupted pyroclastic material and volcanic gases. The craters formed in 2014 occurred in a different location from the previously formed craters of this volcano (Kaneko et al. 2014). The abundance of hydrothermal minerals such as anhydrite and pyrite, as well as the lack of juvenile material in the eruptive products, indicates that the 2014 eruption was essentially phreatic in nature (e.g., Ikehata et al. 2014; Imura et al. 2014; Miyagi et al. 2014; Nakada et al. 2014). Sulfur isotopes have been used as geochemical tracers and temperature indicators for volcanic hydrothermal systems (e.g., Sugiura et al. 1980; Kusakabe et al. 1982; Ossaka et al. 1983; Williams et al. 1990). Kusakabe et al. (1982) reported sulfur isotopic compositions of anhydrite, pyrite, and native sulfur in pyroclastic materials from the phreatic eruption of Mt. Ontake on October 28, 1979. These authors concluded that some of the anhydrite and native sulfur were formed through the disproportionation reaction of sulfite produced by dissolution of magmatic SO_2 into water in the mud reservoir below the crater zone. On the basis of the sulfur isotopic compositions of volcanic gases emitted during the 1979 Ontake eruption, the underground temperature of the crater area in 1979 was calculated to be higher than 250 °C (Ossaka et al. 1983). In this paper, we present sulfur isotopic data of sulfur-bearing minerals and components in the pyroclastic materials from the

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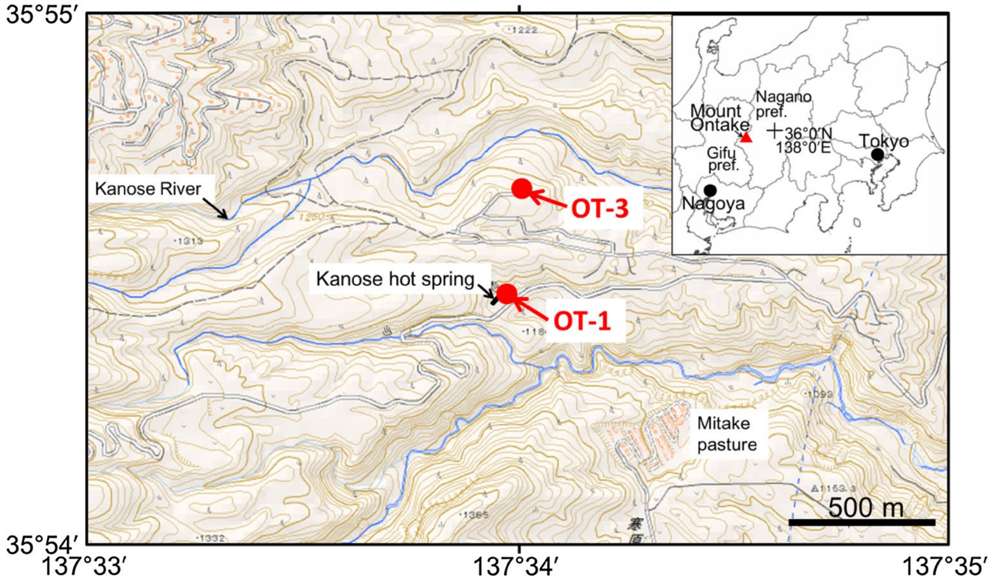

Fig. 1 Locality map of the sampling points for pyroclastic material, OT-1 and OT-3. OT-1 is located at the Kanose hot spring, about 8 km east-north-east of the Jigokudani area of Mt. Ontake, and OT-3 is located about 400 m north of the Kanose hot spring. *Inset* shows the location of Mt. Ontake. These maps are based on the GSI Maps by the Geospatial Information Authority of Japan

2014 Ontake eruption to elucidate the geochemical characteristics of the underlying hydrothermal system.

Sampling locations and analytical methods

Pyroclastic materials from the 2014 Ontake eruption were collected in the morning of September 30, 2014, from two locations near the Kanose hot spring, about 8 km east-northeast of the Jigokudani area of Mt. Ontake: OT-1, a roof of garden shed at the hot spring (Figs. 1, 2), and OT-3, a roadside garbage box located about 400 m north of the hot spring (Figs. 1, 3). The thickness of the pyroclastic material deposited near the Kanose hot spring was about 1 mm (Fig. 4). The collected samples were unaffected by rainwater.

Fig. 2 Photograph of the sampling point of pyroclastic material OT-1, the roof of a garden shed at the Kanose hot spring, in the morning of September 30, 2014

Fig. 3 Photograph of the sampling point of pyroclastic material OT-3, a roadside garbage box, in the morning of September 30, 2014

X-ray diffraction (XRD) analyses were conducted to identify minerals in randomly oriented bulk sample (OT-3) and air-dried oriented clay fractions (<2 μm) separated through elutriation from the OT-3 sample. The clay-fraction samples were also examined with XRD after various treatments were applied (ethylene glycol treatment, HCl treatment, and heating at 500 °C). Thin sections and epoxy

Fig. 4 Photograph of pyroclastic material deposited near the Kanose hot spring, about 8 km east-northeast of the Jigokudani area of Mt. Ontake in the morning of September 30, 2014. The thickness of the pyroclastic material deposited near the Kanose hot spring was about 1 mm

mounts of selected samples were also prepared for petrographic studies and electron probe microanalysis (EPMA).

About 20 g of each of the bulk ash samples (18.9 and 20.9 g of OT-1 and OT-3, respectively) was soaked for 12 h in 800 ml of 18 MΩ deionized water. The supernatant liquid from each sample was then separated from the H_2O solution through filtration using mixed cellulose ester membrane filters with pore sizes of 0.2 µm (Advantec Tokyo, Japan). The H_2O-soaked ash samples were then rinsed with deionized water twice. To ensure oxidation of elemental sulfur, 5 ml of 30 % hydrogen peroxide was added to each of the remaining solutions. These solutions were acidified to a pH of about 3 by adding 2 N HCl. Forty milliliters of 10 % $BaCl_2$ solution was added to the solutions to precipitate barium sulfate. The barium sulfate samples precipitated from the H_2O solutions (water-leached sulfate, H_2O-S) were collected through filtration and rinsed with deionized water. The H_2O-soaked ash samples were treated with 150 ml of 2 N HCl for 2 h at 80 °C. After membrane filtration, the pH of each solution was adjusted to about 2 through the addition of 6 N KOH; 20 ml of 10 % $BaCl_2$ solution was then added to the HCl solutions to precipitate barium sulfate. The barium sulfate samples precipitated from the HCl-treated solutions (hydrochloric acid-leached sulfate, HCl-S) were collected through filtration and rinsed with deionized water. Pyritic sulfide samples (Pyrite-S) were collected from a portion of the acid-treated ash samples (about 2 g) through a density separation method using 1,1,2,2-tetrabromoethane (2.967 g/cm^3). Hundred milliliters of acetone was added to the remaining portion of the HCl-treated ash samples. The acetone solutions were filtered with glass fiber filters (GC-50, Advantec, Tokyo, Japan) and dried in a vacuum chamber at 0.02 MPa at room temperature to obtain evaporated residue of elemental sulfur from the acetone solutions (acetone-leached native sulfur, Native-S).

Sulfur isotopic compositions were determined for the barium sulfate precipitates and the pyritic and elemental sulfur using a helium gas continuous-flow isotope ratio mass spectrometer (CF-IR-MS; Isoprime-EA; Isoprime Ltd., UK) at the University of Tsukuba (see Maruoka et al. 2003a). The samples were weighed in 3.3 mm × 5 mm tin capsules, and 200–300 µg of V_2O_5 was added to each sample. The tin-wrapped samples were introduced into a combustion/reduction quartz tube with an autosampler and were heated at 1070 °C with a helium gas flow of 120 ml/min. The samples were then oxidized with pulses of oxygen. The combustion/reduction tube contained tungstic trioxide to promote the complete oxidation of the samples and pure copper wires to remove excess oxygen and to convert sulfur trioxide to sulfur dioxide. The water produced during combustion was removed by a water trap of magnesium perchlorate. The gases were then introduced into the quartz tube, which contained quartz wool, and held at 890 °C to minimize the oxygen isotope variation of the SO_2, with a SO_2–SiO_2 equilibrium reaction (Fry et al. 2002). Sulfur dioxide and other gases, such as CO_2 and N_2, were separated through gas chromatography. These gases were then introduced with continuous flow of helium into the mass spectrometer through an open-split interface.

The sulfur isotopic compositions are expressed in terms of $\delta^{34}S$ (‰) relative to the V-CDT (Vienna-Canyon Diablo Troilite) standard. The results of three IAEA (International Atomic Energy Agency) silver sulfide standards (IAEA-S-1, −0.3 ‰; IAEA-S-2, +21.80 ‰; and IAEA-S-3, −31.95 ‰; Mayer and Krouse 2004) were compared to constrain the $\delta^{34}S$ values. The isotopic compositions of sulfur were determined to a precision of ±0.1 ‰ (1σ) for 30 µg of sulfur. Precisions were determined based on a combination of the standard deviations from repeated analyses of the samples and standards (Maruoka et al. 2003b); the sample analyses were repeated at least four times.

Results and discussion

Sample description

The collected samples (OT-1 and OT-3) consisted of spherical aggregates of grayish fine volcanic ash (Fig. 5). Stereomicroscopic observation of these samples revealed the presence of clay minerals, hydrothermally altered fragments with sulfate minerals and pyrite, and free crystals of pyrite, feldspar, and silica mineral in both the samples (Fig. 6). No juvenile materials were found in the samples. Alunite, anhydrite, biotite, cristobalite, gypsum,

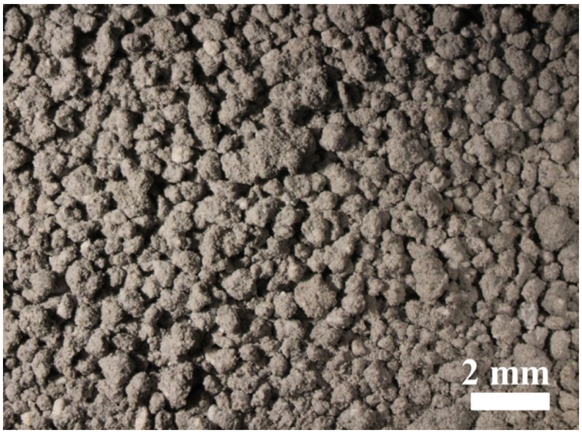

Fig. 5 Photomicrograph of the unwashed pyroclastic material (OT-1) of the 2014 Mt. Ontake eruption under a stereomicroscope

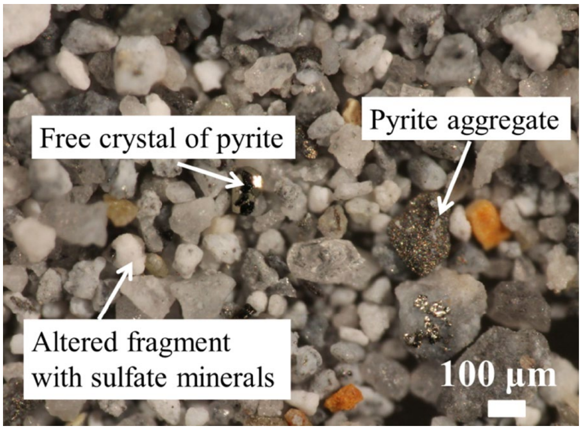

Fig. 6 Photomicrograph of the washed pyroclastic material (OT-1) of the 2014 Mt. Ontake eruption under a stereomicroscope

kaolin minerals, plagioclase, pyrite, pyrophyllite, quartz, and smectite were identified with XRD analyses of the randomly oriented bulk sample (OT-3) and oriented clay fractions of the OT-3 sample (Fig. 7). Ilmenite, orthopyroxene, potassium feldspar, and rutile were also found in OT-3 by EPMA analysis and polarizing microscope observation. These hydrothermal minerals are commonly found in acidic alteration zones of active geothermal areas and sulfur fumaroles (e.g., Utada 1980; Hedenquist et al. 1996). Cristobalite and kaolin minerals are low-temperature (i.e., less than 200 °C) acid-stable minerals, whereas pyrophyllite and rutile are relatively high-temperature (200–300 °C) acid-stable minerals (Hedenquist et al. 1996). Most of the hydrothermal minerals of the Mt. Ontake 2014 eruptive products were also reported from pyroclastic materials of the 1979 Mt. Ontake phreatic eruption (e.g., Sugiura et al. 1980; Ossaka et al. 1983), which suggests similarity in the temperature ranges of the hydrothermal systems of both eruptions.

Sulfur isotopic characteristics

Sulfur isotopic compositions of sulfur from the water-leached sulfate (H_2O-S), hydrochloric acid-leached sulfate (HCl-S), acetone-leached native sulfur (Native-S), and pyrite (Pyrite-S) from the collected samples (OT-1 and OT-3) are presented in Table 1 and Fig. 8.

The $\delta^{34}S$ values of H_2O-S of OT-1 and OT-3 are +15.53 ‰ and +15.22 ‰ and are similar to those of the HCl-S results of +15.92 ‰ (OT-1) and +16.04 ‰ (OT-3), respectively. The sulfur isotopic compositions of the HCl-S for the 2014 Ontake phreatic eruption are slightly higher than those of the HCl-S from the 1979 Ontake phreatic eruption ($\delta^{34}S = +8.3$ ‰ to +14.0 ‰; Kusakabe et al. 1982). The Native-S of OT-1 and OT-3 has $\delta^{34}S$ values of −3.86 ‰ and −3.77 ‰, respectively, and is within the reported range of the Native-S from the 1979 eruption ($\delta^{34}S = -5.3$ ‰ to −1.8 ‰; Kusakabe et al. 1982). The $\delta^{34}S$ values of Pyrite-S of OT-1 and OT-3 are −5.24 ‰ and −5.17 ‰, in the same range as those of the Pyrite-S from the 1979 eruption ($\delta^{34}S = -6.4$ ‰ to −5.4 ‰; Kusakabe et al. 1982). There are no significant differences in sulfur isotopic compositions of H_2O-S, HCl-S, Native-S, or Pyrite-S between OT-1 and OT-3. Isotopic fractionation between reduced sulfur and oxidized sulfur species occurs during disproportionation of magmatic SO_2 in the presence of water through the following reaction (Holland 1965),

$$4SO_2 + 4H_2O = 3H_2SO_4 + H_2S, \quad (1)$$

which produce isotopically light sulfides and heavy sulfates (Ohmoto and Rye 1979; Rye 2005). The sulfur isotopic values of pyroclastic materials from the 2014 Ontake eruption can be explained by the reaction above occurring in the underlying hydrothermal system (mud reservoir), as proposed for the 1979 Ontake eruption and volcanic activities in other regions (e.g., Kusakabe et al. 1982; Williams et al. 1990). Also as proposed for the case of the 1979 Ontake eruption (Kusakabe et al. 1982), some native sulfur of the 2014 eruptive products was probably formed in the presence of water in the mud reservoir beneath Mt. Ontake through a different disproportionation reaction (Holland 1965),

$$3SO_2 + 2H_2O = 2H_2SO_4 + S. \quad (2)$$

Kusakabe et al. (1982) also suggested that some native sulfur with heavier $\delta^{34}S$ values (−3.6 ‰ to −1.8 ‰) than the others (−5.3 ‰ to −5.1 ‰) was formed by oxidation of H_2S around fumaroles. Native sulfur with

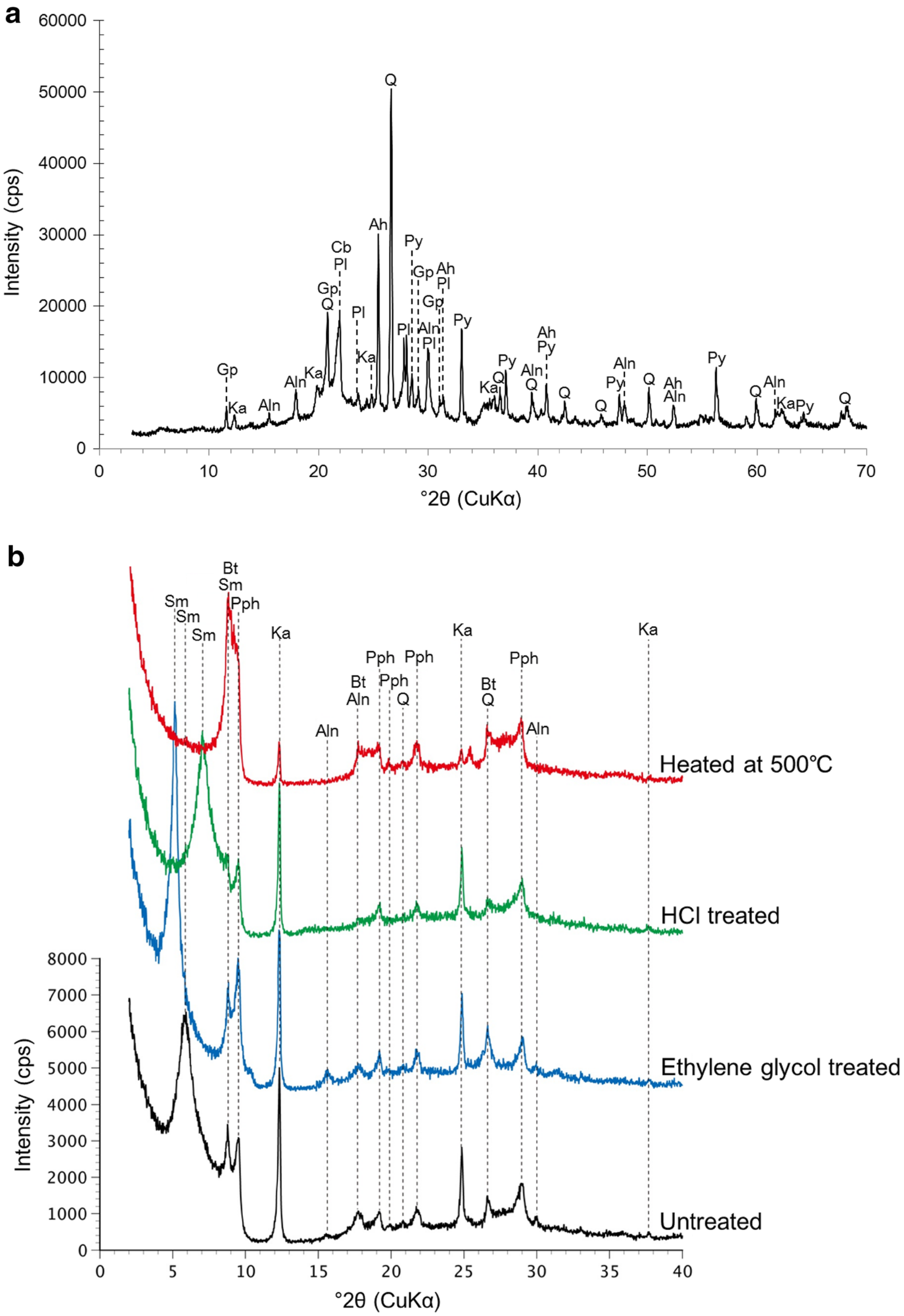

Fig. 7 X-ray diffraction (XRD) patterns of **a** randomly oriented bulk pyroclastic material (OT-3) and **b** oriented clay fractions (<2 μm) separated through elutriation of the OT-3 sample from the 2014 Mt. Ontake eruption. The clay-fraction samples were examined with XRD after various treatments (ethylene glycol treatment, HCl treatment, and heating at 500 °C). Note that the smectite peak of the untreated clay fraction was shifted after the ethylene glycol, HCl, and heating treatments. *Abbreviations* for minerals: *Aln* alunite, *Ah* anhydrite, *Bt* biotite, *Cb* cristobalite, *Gp* gypsum, *Ka* kaolin minerals, *Pl* plagioclase, *Py* pyrite, *Pph* pyrophyllite, *Q* quartz, *Sm* smectite

Table 1 Sulfur isotopic values and concentrations of sulfur species of the pyroclastic materials of the 2014 Mt. Ontake eruption

Sample	Sulfur species	S (wt%)	$\delta^{34}S$ (‰)	±1σ (‰)
OT-1	Water-leached sulfate (H_2O-S)	0.50	+15.53	0.10
OT-1	Hydrochloric acid-leached sulfate (HCl-S)	0.62	+15.92	0.08
OT-1	Acetone-leached native sulfur (Native-S)	0.22	−3.86	0.07
OT-1	Pyrite (Pyrite-S)	1.79[a]	−5.24	0.24[b]
OT-3	Water-leached sulfate (H_2O-S)	0.54	+15.22	0.09
OT-3	Hydrochloric acid-leached sulfate (HCl-S)	0.58	+16.04	0.07
OT-3	Acetone-leached native sulfur (Native-S)	0.22	−3.77	0.08
OT-3	Pyrite (Pyrite-S)	1.12[a]	−5.17	0.37[b]

[a] Some pyrite grains attached to lighter minerals (e.g., silicate minerals) might not have precipitated during density separation; therefore, the values should be considered as minimum sulfur concentrations

[b] The errors for pyritic sulfur in this study were larger than the precision determined for other samples; however, such larger errors likely originated from isotopic heterogeneity

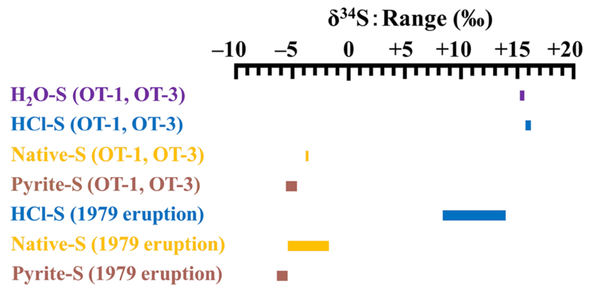

Fig. 8 Diagram showing variations in $\delta^{34}S$ values of sulfur from water-leached sulfate (H_2O-S), hydrochloric acid-leached sulfate (HCl-S), acetone-leached native sulfur (Native-S), and pyrite (Pyrite-S) for the pyroclastic materials of the OT-1 and OT-3 samples from the 2014 Mt. Ontake eruption compared to those of sulfur from hydrochloric acid-leached sulfate (HCl-S), acetone-leached native sulfur (Native-S), and pyrite (Pyrite-S) for the pyroclastic materials from the 1979 Mt. Ontake eruption (Kusakabe et al. 1982)

a similar sulfur isotopic composition ($\delta^{34}S = -2.6$ ‰) was reported around fumaroles related to the 1979 Mt. Ontake eruption (Ossaka et al. 1983). If sulfur isotopic equilibrium between anhydrite and pyrite is assumed, the observed sulfur isotope fractionations of the 2014 eruption correspond to sulfur isotopic equilibration temperatures from 270 to 281 °C calculated using the following equation (Ohmoto and Rye 1979; Ohmoto and Lasaga 1982),

$$1000 \ln \alpha_{\text{anhydrite}-\text{pyrite}} = 6.063\left(10^6/\text{T}^2\right) + 0.56\ (\text{T in K}), \tag{3}$$

where $\alpha_{\text{anhydrite-pyrite}}$ represents the isotope fractionation factor between anhydrite and pyrite.

This temperature range is consistent with those inferred from the hydrothermal mineral assemblage (e.g., pyrophyllite and rutile) of the 2014 pyroclastic materials (200–300 °C) (Hedenquist et al. 1996), as well as the underground temperature of the 1979 crater area calculated based on the sulfur isotopic compositions of volcanic gases related to the 1979 Ontake eruption (more than 250 °C; Ossaka et al. 1983). These ranges are lower than the sulfur isotopic equilibration temperatures of the 1979 eruption (314–394 °C) calculated based on the sulfur isotopic values of HCl-S and Pyrite-S (Kusakabe et al. 1982) with Eq. 3. Sugiura et al. (1980) and Kusakabe et al. (1982) suggested that these temperatures are unrealistically high for the underground mud reservoir of the 1979 eruption because the clay minerals would be unstable at such high temperatures. They attributed this discrepancy to the incorporation of sulfate minerals with lower sulfur isotopic values [e.g., a gypsum sample ($\delta^{34}S = +5.1$ ‰) from a fumarolic area of Mt. Ontake] into the underground materials.

Conclusions

The pyroclastic material of the 2014 Mt. Ontake eruption was composed of altered volcanic fragments, alunite, anhydrite, biotite, cristobalite, gypsum, ilmenite, kaolin minerals, native sulfur, orthopyroxene, plagioclase, potassium feldspar, pyrite, pyrophyllite, quartz, rutile, and smectite. Most of these minerals were likely derived from the acidic alteration zones of Mt. Ontake. The absence of juvenile material in the eruptive products indicates that the eruption was phreatic. The difference in sulfur isotopic values between anhydrite and pyrite from the 2014 eruption indicates isotopic equilibration temperatures ranging from 270 to 281 °C. This temperature range is consistent with that inferred from the hydrothermal mineral assemblage (e.g., pyrophyllite and rutile) of the 2014 pyroclastic materials (200–300 °C). With the exception of the sulfur isotopic compositions of anhydrite, which may have been altered by the incorporation of sulfate minerals in a fumarolic area with lower sulfur isotopic values into the underground materials during the 1979 eruption (Sugiura et al. 1980; Kusakabe et al. 1982), no significant differences in the mineral assemblages or sulfur isotopic compositions of the pyroclastic materials were observed between the 2014 and 1979 Ontake phreatic eruptions, which indicates geochemical similarity in the hydrothermal systems below the crater before each of these eruptions.

Authors' contributions

IK carried out the sample collection, sample description and drafted the manuscript. MT performed the sulfur isotope analysis of the samples and helped draft the manuscript. Both authors read and approved the final manuscript.

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Competing interests

The authors declare that they have no competing interests.

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