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To cite this article: I Kusmartini *et al* 2017 *J. Phys.: Conf. Ser.* **860** 012005

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# Elemental characterization of Mt. Sinabung volcanic ash, Indonesia by Neutron Activation Analysis

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**Abstract.** Mount Sinabung is a volcano located in North Sumatera, Indonesia which has been recorded not erupted since 1600. However in 2013 it has been erupted and cause of black thick smog, rain sand and volcanic ash. Volcanic ash containing trace elements material that can be utilized in various applications but still has potential danger of heavy metals. In order to obtain an elemental composition data of volcanic ash, the characterization of volcanic ash were carried out using Neutron Activation Analysis. The volcanic ash was taken from Mt. Sinabung eruption. Samples were irradiated at the rabbit system in the reactor G.A Siwabessy facilities with neutron flux  $\sim 1013 \text{ n.cm}^{-2}\cdot\text{s}^{-1}$  and then counted using HPGe detector. Method validation was carried out by SRM NIST Coal Fly Ash 1633b and NIST 2711a Montana II Soil with recovery values were in the range of 96–108% and 95–106% respectively. The results showed that major elements; Al, Na, Ca and Fe, concentrations were 8.7, 1.05, 2.98 and 7.44 %, respectively, minor elements K, Mg, Mn, Ti, V and Zn were 0.87%, 0.78%, 0.18%, 0.62%, 197.13 ppm and 109.35 ppm, respectively, heavy metals; As, Cr, Co and Sb, contents were 4.48, 11.75, 17.13 and 0.35 ppm, respectively while rare earth elements such as Ce, Eu, La, Nd, Sm, Yb were 45.33, 1.22, 19.63, 20.34, 3.86, and 2.57 ppm respectively. The results of the elemental contents of volcanic ash that has been obtained can be used as the scientific based data for volcanic material utilization by considering the economic potential of elements contained and also the danger of the heavy metals content.

## 1. Introduction

Mount Sinabung is a stratovolcano, located in Karo Regency, North Sumatra Province of Indonesia. It is a solitary volcano that has a single peak, and classified as B -Type, because since the 1600's there was no record of eruptive activity [1]. But on November 24, 2013, Mount Sinabung were erupted and reach the highest alert level (level 4) since it spat out black and thick smog, followed by rain sand and volcanic ash which covered thousands of hectares of farmer crops under the radius of six kilometers [2]. Volcanic ash is fragments of magma and consists of mineral, volcanic glass and also a material containing high silica and aluminum [2,3]. The composition of particles from volcanic eruptions should reflect the matrix composition of the magma.  $\text{SiO}_2$  contents of 40–55 wt% and CaO plus  $\text{K}_2\text{O}$  between 4 and 6 wt% match the composition of basaltic to basalt andesitic magma [4].

Related of that there are several studies of the characterization of volcanic ash., F. Ruggieri, *et al* (2012) had been researched mineral compositions of volcanic ash samples of Chaiten volcano, Chile Southern, such as  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ , MnO, MgO, CaO,  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ ,  $\text{TiO}_2$  were 72.06, 14.66, 1.88, 0.07, 0.6, 1.99, 4.18, 2.89, 0.2 % respectively [5]. These materials in the ash can provide important information on the nature of magma because chemical compositions of magma usually show distinct features of each volcano and because the assemblages and compositions of minerals



reflect their host [6]. The other study had been done by Bonnie Canon, *et al* (2012) that found of trace elements in volcanic ash from Mount Merapi, such as As, Co, Cr, Sb, V, Zn [7]. General features of the studied volcanic ashes from the Puna in Northwestern Argentina by F Ruggieri *et al* (2010) also obtained rare earth elements such as La, Ce, Nd, Sm, Eu, Yb [8]. The rare earth elements (REEs) or rare earth metals (REMs) as defined by IUPAC, represent a group of 17 transient metals. They comprise scandium, yttrium and further 15 elements called lanthanides, ranging from the most abundant cerium and lanthanum to the less frequent lutetium. The natural abundances of REEs greater than their name would suggest, with several elements being present at an average content of 30 mg/kg on the Earth's crust [9]. Therefore explosive eruptions are capable of causing significant environmental repercussions, affecting simultaneously many cycles in the Earth's system, impacting on water, soil and surface sediments. An important reflection of volcanic activity is the natural contribution of chemical elements [10].

Neutron Activation Analysis (NAA) is a sensitive analytical technique which useful for performing both qualitative and quantitative multi-elemental analysis of major, minor and trace elements in samples from almost every field of scientific or technical interest. Particularly advantage is taken of the fact that the samples do not have to undergo any chemical treatment, non destructive which in many materials belong to the major matrix components. It enables the observation of trace elements, often at amounts in the order of microgram to nanogram. The high selectivity of gamma-ray spectrometry allows for simultaneous determination of many radionuclides [22]. NAA is an established technique which can be used for the measurement of trace element levels in various matrices. It involves neutron activation of the different trace elements in the sample and measurement of the radioactivity of each radioisotope formed in order to measure the individual concentrations of elements. NAA facilitates of elements in samples of different matrices especially of environmental origin. It is one of the most mature analytical methods currently used and yet remains highly compete with others in term of accuracy, detection limits and multi elemental capabilities [11,12].

## 2. Methodology

### 2.1. Sample Collection and Preparation

The samples of ash were collected in yard of residential dwelling as far as 6 km of the volcano. These sites were located in mountain side of Sinabung Karo Regency, North Sumatra Province. Volcanic ash samples were taken about 1 kg of the 2013 eruption. The volcanic ash samples were dried at temperature 45–60 °C using infrared lamps. After it dried reduction of particles size were done using electrical pulverisette and 200 mesh sieve to get  $\leq 74\mu\text{m}$  particles size of volcanic ash.

### 2.2. Standard Preparation

About 25–40 mg of volcanic ash were weighted and put into 0.3 mL polyethylene vials. Certified reference material from National Institute of Standards and Technology (NIST), SRM NIST 1633b and SRM NIST 2711a Montana II Soil were used to validate the method. Mix standards for relative method were prepared and placed in the same size vials. Preparation of NAA standards was carried out by pipetting 100  $\mu\text{L}$  of ICP multi element standard (E-Merck) then dripped in the polyethylene vial. The vial dried under the infrared lamp then sealed by heating. Weights of elements in the prepared standards were shown in Table 1.

### 2.3. Irradiation and Gamma Measurement

The samples, certified reference materials and standards were irradiated in the rabbit system of The Multi-Purpose Reactor G.A Siwabessy in Serpong, Banten Province of Indonesia with neutron flux  $\sim 10^{13} \text{ n.cm}^{-2}.\text{s}^{-1}$ . The condition of irradiation were shown in Table 2. After cooled the samples dan standard measured using a spectrometer gamma ray, HPGe detector manufactured by Canberra. The spectrum analysis was carried out using GENIE 2000.

**Table 1.** The weight of elements for preparation NAA standards

Elements	Weight (ug)
Al	21
Na	20.98
Ca	101.2
Fe	49.9
K	21
Mg	21
Mn	1
Ti	10
V	1.01
Zn	10
As	9.8
Cr	1.01
Co	1
Sb	1
La	0.1
Sm	0.1
Eu	1
Yb	1
Ce	1
Nd	1

**Table 2.** Irradiation conditions

Element	Half Time	Irradiation Time	Delay/Cooling	Counting Time
Al, Ca, Ti, Mn, V, Mg	Short-lived radionuclide	1 minute	1 minute	200 seconds
Na, K, As, Sm, La	Medium-lived radionuclide	10 minutes	1 day	2000 seconds
Fe, Co, Cr, Sb, Eu, Yb, Ce, Nd, Zn	Long-lived radionuclide	1 hour	1 week	4000 seconds

Reliability of volcanic ash analysis results depends on instruments performance as well as personal capability and was proven by its accuracy. Accuracy can be defined as a measure of the closeness of the results with its true value while precision can be considered as a measure of the repeated and reproducibility of a result. Accuracy can be stated as % recovery. Recovery is the ratio between measurement result with certificate value [13], expressed by the formula (1):

$$\%recovery = \frac{value_{analyt}}{value_{certificate}} \times 100\% \quad (1)$$

The accuracy of method were determined by comparing results of the SRM 1633b and SRM NIST 2711a Montana II Soil with its certificate value respectively. Acceptable criteria for accuracy were based on AOAC *guidelines for single laboratory validation* shown in Table 3 [13].

**Table 3.** Acceptable criteria for accuracy [13]

Concentration	% Recovery
100%	98-101
10%	95-102
1%	92-105
0,1%	90-108
0,01%	85-110
10 ppm	80-115
1 ppm	75-120
10 ppb	70-125

### 3. Result and Discussion

Validation of NAA was carried out using NIST Coal Fly Ash 1633b and NIST 2711a Montana II Soil Standards Reference Material (SRM). Table 4 showed the results of measurement of SRM NIST Coal Fly Ash 1633b with %recovery were in the range of 96–108%. These values were in the %recovery of acceptance criteria for 0.1% concentration 90–108% [13]. While in the Table 5 showed the results of measurement of 2711a Montana II Soil by NAA method with %recovery were in the range of 95–106%. These values were also in the %recovery range of acceptance criteria for 0.1% concentration that is 90–108% [13]. It might be said that the results for Coal Fly Ash 1633b and 2711a Montana II Soil are in good agreements with the recommended certified value and therefore the NAA method was quite accurate for determining the elemental contents of similar matrices of samples that being analyzed.

In this studies NAA method was applied to determine the concentrations of 20 elements (Al, Na, Ca, Fe, K, Mg, Mn, Ti, V, Zn, As, Cr, Co, Sb, La, Sm, Eu, Yb, Ce and Nd) shown in Table 6 and the figure of gamma rays spectrum were shown in Figure 1. The results of this study were compared to those found by other published studies illustrating the elemental mean concentration levels in volcanic ash of other regions of some prominent states of Europe, Africa, South America and Asia regions [12]. The mean value of Al concentration in Mt. Sinabung volcanic ash, Indonesia were found to be similar with permian Trassic's China [15], while the element of Ca 3 times lower than found in Mt. Shihan, Jordan [16]. Sodium contents of Mt. Sinabung volcanic ash were three times lower than in volcanic

**Table 4.** Elemental concentration (ppm) in NIST Coal Fly Ash 1633b Standard

Element	Unit	Measured (ppm)	Certificate (ppm)	% Recovery
Al	%	14.4 ± 0.2	15.05 ± 0.27	96%
Ca	%	1.5 ± 0.2	1.51 ± 0.06	100%
Fe	%	7.8 ± 0.2	7.78 ± 0.04	100%
K	%	1.9 ± 0.03	1.95 ± 0.03	100%
Na	%	0.2 ± 0.0008	0.201 ± 0.003	100%
Mg	%	0.46 ± 0.11	0.48 ± 0.008	96%
Ti	%	0.7 ± 0.06	0.79 ± 0.014	98%
V	%	0.03 ± 0.001	0.03 ± 0.003	100%
Ce	ppm	190 ± 1.24	190	100%
Cr	ppm	200 ± 6.35	198 ± 4.7	101%
Co	ppm	48 ± 0.5	50	97%
La	ppm	94 ± 1.47	94	100%
Sc	ppm	40 ± 0.1	41	99%
Sm	ppm	20 ± 0.2	20	100%
Zn	ppm	227 ± 63	210	108%

**Table 5.** Elemental concentration (ppm) in NIST 2711a Montana II Soil Standard

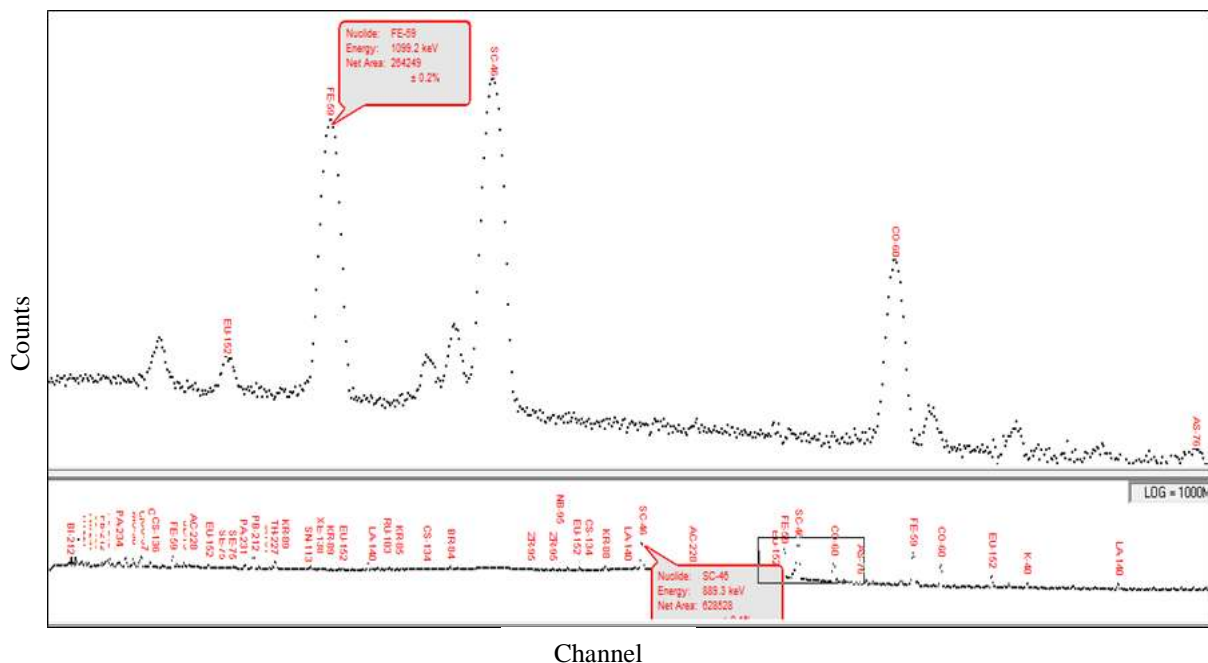
Element	Unit	Measured (ppm)	Certificate (ppm)	%Recovery
Al	%	6.64 ± 0.15	6.72 ± 0.06	99%
Ca	%	2.35 ± 0.13	2.42 ± 0.06	97%
Fe	%	2.83 ± 0.04	2.82 ± 0.04	100%
K	%	2.68 ± 0.06	2.53 ± 0.1	106%
Mg	%	1.07 ± 0.16	1.07 ± 0.06	100%
Mn	%	0.06 ± 0.0016	0.06 ± 0.0018	100%
Na	%	1.21 ± 0.003	1.2 ± 0.01	102%
Ti	%	0.32 ± 0.04	0.31 ± 0.008	99%
As	ppm	107 ± 4	107 ± 5	100%
Ce	ppm	70 ± 5.7	70	100%
Co	ppm	9.89 ± 0.2	9.89 ± 0.18	100%
Cr	ppm	52.7 ± 1.21	52.3 ± 2.9	100%
Eu	ppm	1.1 ± 0.7	1.1 ± 0.2	100%
La	ppm	37 ± 1	38 ± 1	98%
Sc	ppm	8.4 ± 0.1	8.5 ± 0.1	99%
Sm	ppm	5.8 ± 0.22	5.9 ± 0.28	98%
V	ppm	76.6 ± 5.7	80.7 ± 5.7	95%
Zn	ppm	424 ± 10	414 ± 11	102%

ash of Mt. Puna, Argentina [14], while concentration of Fe in Mt. Sinabung volcanic ash, Indonesia and Mt. Grímsvötn Denmark were almost have similar value. Further, Mg in Mt. Sinabung's volcanic ash, Indonesia were has concentration 4 times lower than Mg concentration of Mt. Shihan's, Jordan. However, concentration of Mn in Mt. Sinabung's volcanic ash was the highest among others.

The value of trace elements; V, Zn, As, Cr, Co and Sb concentrations and its comparisons with other results were also shown in Table 6. The content of V and Zn in Mt. Sinabung's volcanic ash was the highest compared with other results, but if it compared with Cordón Caulle Volcanic Complex, Argentina both Zn concentrations tend to have similar value. The mean value of As was 9 times lower than in Chaitén Volcano Chile and 4 times higher than in Alpine Anthering volcano Austria. For Cr the mean value in Indonesia was 23 times lower than Mt. Shihan's, Jordan and 2 times higher than Puna Volcano, Argentina. Concentration of Co was three times lower than in Mt. Shihan's Jordan however Sb contains in Mt. Sinabung Indonesia has the smallest concentration among other results. The particular attention should be given to the concentration of potential pollutants semi-volatile elements; As and Sb, which can be associated considering their affinity of fluid or gas phases or forming sulphate or halide volatile compounds at magmatic temperatures, being adsorbed onto ash particles. These elements especially As are potential toxic trace elements for the environmental [14]. Determination results of rare earth elements (La, Sm, Eu, Yb, Ce and Nd) in volcanic ash were also shown in Table 6. The concentration of La in Mt. Sinabung, Indonesia was three times lower than in Alpine Anthering Volcano Austria also for the element of Sm was three times lower than in Eyjafjallajökull Volcano, Iceland. The mean value of Eu in Mt. Sinabung, Indonesia and Mt. Shihan, Jordan were found to be similar. For Ce in Mt. Sinabung, Indonesia was three times lower than in Alpine Anthering Volcano Austria however with Chaitén Volcano, Chili and Mt. Shihan, Jordan the concentrations were in the same range. Concentration of Nd in Mt. Sinabung, volcanic ash was two times lower than found in Alpine Anthering Volcano Austria, Cordón Caulle Volcanic Complex, Argentina and Eyjafjallajökull Volcano Iceland but four times higher than Permian-Triassic, China. Rare earth elements and their compounds, due to the unique magnetic, electric and optical properties are used in many kinds of industrial products such as, the photo luminescence materials, magnetic alloys, catalysts for petroleum refining, carrier materials of automobile exhaust catalysts and high-refractive index low-dispersion optical glasses. Moreover,  $\text{LaCl}_3$  is added to the chemical fertilizers to increase the yields of crop [9].

The differences concentration of elements for country to each volcanic ash depend on the physical fractionation of magma during the explosive events and the subaerial transport by wind currents could have changed the original chemical composition of volcanic ash [19]. These results support earlier statement that there is a difference in the concentration of trace elements. This could be due to the geological conditions and geochemical factors such as hydrothermal fluids have been reported to be responsible for the enrichment of trace elements. Beside the trace elements available these ashes are also provided with macro and micronutrients (Mn, Mg and Fe) evidencing the fertilising potential of the ashes. Although the total contributions of such elements are relatively low in ashes, their deposition could provide an external critical nutrient source in low nutrient areas [6].

Many other elements studied are potentially toxic trace elements (PTTEs) and some of them are included in the drinking water guidelines due to their potential toxicity even at very low concentrations and must be especially monitored in the environmental assessment of these ash fall deposits. Human and animal health effects by expose of ash, linked to respiratory illness, is closely associated with particle size, which are especially anticipated for diameters  $\leq 10 \mu\text{m}$  because aerodynamically fine particles successfully negotiate the curves of the throat and are carried to the lungs. An aircrafts operations (especially commercial jets) is threatened by volcanic cloud encounters, which a variety of hazards can exist such as airborne very fine ash that enters and melts in jet turbines and causing failure [20]. Volcanic ash emissions are episodic events that contribute approximately an order of magnitude less silicate particles to the atmosphere on average that contributed by the mineral dust cycle. To place this global context, the total mass of global emissions of fine ash may be estimated from historic records of eruption frequency and assumptions on the proportion of fine ash generated by different styles of explosive volcanism [21].



**Figure 1:** Gamma-rays spectrum from software GENIE 2000

**Table 6.** Elemental concentrations in volcanic ash in this study and other regions

Element	Unit	Present study, Sinabung Volcanic Indonesia 2013	LOD	<i>n</i>	Cordón Caulle Volcanic Complex Argentina 2011 <sup>a</sup>	Chaitén Volcano Chile 2008 <sup>b</sup>	Shihan Volcano Jordania 2007 <sup>c</sup>	Grimsvötn Volcano, Denmark 2011 <sup>d</sup>	Eyjafjallajökull Volcano Iceland 2010 <sup>e</sup>	Permian-Triassic Boundary China 2015 <sup>f</sup>	Alpine Anthering Volcano, Austria 2003 <sup>g</sup>	Puna Volcano Argentina 2010 <sup>h</sup>
Al	%	8.70 ± 2.26	1.E-03	3	7.56	3.88	4.08	4.68		7.89	5	3.29
Na	%	1.05 ± 0.12	2.E-06	5	3.88	1.55	1.40	0.81		0.05	0.15	1.45
Ca	%	2.98 ± 0.04	1.E-02	3	1.95	1.42	7.02	0.5		0.61	1.52	0.63
Fe	%	7.44 ± 0.5	2.E-06	5	3.37	0.66	3.97	7.7		0.89	1.5	0.28
K	%	0.87 ± 0.13	2.E-04	5	2.73	1.19	0.50	1.19		2.61	0.27	1.86
Mg	%	0.78 ± 0.11	3.E-03	3	0.55	0.36	4.17	1.8		1.34	1.2	0.09
Mn	%	0.18 ± 0.02	1.E-05	3	0.09	0.05	0.13				0.03	0.06
Ti	%	0.62 ± 0.09	1.E-03	3	0.38	0.12	1.04			0.34	0.46	0.06
V	ppm	197 ± 5.12	1.E-01	3	33	13.8	176				27	6.6
Zn	ppm	109 ± 20.5	2.E-03	5	104	51.2	94				50	39.8
As	ppm	4.48 ± 0.79	2.E-03	3	15	38.4					<1	5.75
Cr	ppm	11.8 ± 2	2.E-04	5	9.1	19	281				10	4.49
Co	ppm	17.1 ± 1.07	5.E-02	5	6.25	2.57	42				2.38	0.82
Sb	ppm	0.35 ± 0.004	2.E-06	2	1.02	1.51					0.44	1.13
La	ppm	19.6 ± 2.2	5.E-03	5	37	24.4	25		40.9	3.77	63.6	14.5
Sm	ppm	3.86 ± 0.43	7.E-04	5	9.43	2.83	4.8		11.4	2.25	9.84	5.26
Eu	ppm	1.22 ± 0.06	9.E-06	5	1.82	0.66	1.2		3.27	0.83	2.42	0.79
Yb	ppm	2.57 ± 0.79	1.E-02	5	6.86	1.44	1.1		4.78	4.36	3.09	1.77
Ce	ppm	45.3 ± 5.27	2.E-04	5	88	46.5	48		93	9.81	135	33.7
Nd	ppm	20.3 ± 1.84	6.E-04	5	44.8	16.5	17		48.6	4.7	53.1	12.7

<sup>a</sup> Daga R et al., 2014 [14].<sup>b</sup> Ruggieri F et al., 2012 [6].<sup>c</sup> Ibrahim KM et al., 2014 [16].<sup>d</sup> Lieke KI et al., 2013 [4].<sup>e</sup> Tepe N and Bau M. Et al., 2014 [18].<sup>f</sup> Liao Z et al., 2016 [15].<sup>g</sup> Huber H et al., 2003 [17].<sup>h</sup> Ruggieri F et al., 2010 [8].



#### 4. Conclusions

The characterization results showed that Mt. Sinabung volcanic ash contains major elements; such as Al, Na, Ca and Fe with their concentrations were 8.7, 1.05, 2.98 and 7.44 %, respectively, minor elements; K, Mg, Mn, Ti, V and Zn with their concentration were 0.87, 0.78, 0.18, 0.62 %, 197.13 and 109.35 ppm, respectively while heavy metals As, Cr, Co and Sb were 4.48, 11.75, 17.13 and 0.35 ppm, respectively and rare earth elements Ce, Eu, La, Nd, Sm, Yb were respectively 45.33, 1.22, 19.63, 20.34, 3.86, and 2.57 ppm. The differences concentration of volcanic ash of country to each other depend on the physical fractination of magma during the explosive events and the subaerial transport by wind currents that could have changed the original chemical composition of volcanic ash. The results support earlier statement that there is a difference in the concentration of trace elements that could be due to the geological conditions and geochemical factors which have been reported to be responsible for the enrichment of trace elements. Related to that, the results of elemental contents of volcanic ash obtained can be used as the scientific based data for volcanic material utilization by considering the economic potential of rich elements and its danger of the heavy metal contents.

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