



# Spatial Variation of Rare Earth Elements (REEs) in Agricultural Soils Collected from Different Geographical Regions in Vietnam

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## Abstract

Rare earth elements (REEs) exist commonly in minerals, such as carbonates, silicates, fluorides, and phosphates. REEs are also found in coals, animals, algae, and plants which were proven to increase the growth, development, yield, and quality of crops. In the present study, a preliminary assessment of REEs contents in soils collected from different areas in Vietnam was performed using the methods of Fe(OH)<sub>3</sub> and Mg(OH)<sub>2</sub> co-precipitation to exclude interferences from the sample matrix before analyzing REEs on ICP-MS system. The contents of REEs (mg kg<sup>-1</sup>) were in descending of Ce > Nd > La > Pr > Sm > Gd > Dy > Tb > Er > Yb > Lu. The light REEs contents were always higher than the heavy REEs, accounting for more than 70% of the total contents. In addition, the REEs also differed in total contents between areas. The contents of REEs were as follows Northwest > Northeast > Central Highland > Southeast. These differences could be related to influences during the natural weathering processes for soil formation and human activities such as mining and cultivation via fertilizers.

**Keywords:** Rare earth elements, REE, Co-precipitation, Agricultural soil, Geography, ICP-MS.

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## Introduction

The term rare earth elements includes 15 lanthanides with yttrium and scandium [1]. They are divided into two groups: light REEs – LREE (La, Ce, Pr, Nd, Pm, Sm, and Eu) and heavy REEs – HREEs (Y, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu) [1-4]. In the Earth's crust, the REEs content accounts for 0.01 - 0.02%, approximately with the content of Cu, Ni, Zn, and Pb, which are higher than Sn, Co, Ag and Hg. The content of LREEs is about 200-fold higher than the HREEs [3]. In the environments, REEs exist in more than 270 minerals as carbonates, silicates, fluorides,

and phosphates. They are also found in algae, plants, animals, coals and rocks [2, 5]. The REEs in soils originate from the weathering of parent rocks such as granite, basalt, and acid igneous rock [3]. In addition, the deposition of REEs from the atmosphere and agricultural activities using fertilizers are important causes for REEs to enter the soil [6]. The total content of REEs in the soil depends on the type and composition of the parent rock, e.g., clay content [7], acidity or basicity (pH change) [3], carbonate (CO<sub>3</sub><sup>2-</sup>) [8], total organic carbon (TOC) [9], Al, Fe, Mn, and

redox potential [10] which affects the physicochemical properties of the soils. The distribution of REEs is uneven among regions in the world, mainly in China, Vietnam, the United States [11], etc., although the REEs are quite high content.

The effects of REEs on crops have been explored since the 30s, improving crop growth, development, yield, and quality, which are often added to fertilizers as microelements [12, 13]. For instance, in rice, 0.05 - 1.5 mgL<sup>-1</sup> of La<sup>3+</sup> increased yield and the dry weight of roots, while Ce<sup>3+</sup> stimulated growth and improved the chlorophyll content [14, 15]. In wheat leaves, the used individual REEs and LREEs increased chlorophyll-a and chlorophyll-b content. Nitrate REEs increased chlorophyll (29.6%), germination (8-9%), and the content of 7 amino acids from 2 to 21%. Moreover, nitrate REEs also increased the content of amino acids in soybean (8%); in maize (15.2%), enhanced CO<sub>2</sub> assimilation to 35.8 - 79.8%, and raised photosynthesis from 11.5 - 31.2%. REEs also increased the fiber content of cotton (5-12%), potato (5.7%), rubber (8 - 10%), soybean (8 - 9%), and maize (8.5 - 103%) [13].

Currently, Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) is considered the most common one for the simultaneous determination of REEs at the trace level. However, a complex matrix of soils coupled with the presence of high contents of Al and Fe can affect the analytical accuracy without matrix elimination including those elements. Taicheng et al. have applied the co-precipitation technique using Fe(OH)<sub>3</sub> and Ti(OH)<sub>4</sub> for analyzing REEs on ICP-MS [16]. Depending on the sample compositions, different sample preparation could be applied to get accuracy, for instance, using an acid mixture of HF/HNO<sub>3</sub>, HF/HClO<sub>4</sub>, HF/H<sub>2</sub>SO<sub>4</sub>, HF/HBr/HNO<sub>3</sub>, HF/HNO<sub>3</sub>/HClO<sub>4</sub>. An alternative sample preparation to acid

digestion is an alkaline fusion with LiBO<sub>2</sub>/Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, commonly applied to stable mineral samples to digest the silicate matrix [17].

Vietnam is a country with large reserves of minerals rich in REEs such as monazite, fluor carbonate, and rare earth mines, e.g., Nam Xe, Dong Pao, and Yen Phu, which can contribute to the increase of REEs content in plants. There is a lack of research on the distribution of REEs in agricultural soils in Vietnam although they are applied in many fields and could be exposed by mining activities. In the present study, we aimed to assess the spatial distribution of REEs in different soils from areas characterized by different topographical conditions, climate, and farming processes in Vietnam, using the co-precipitation technique of Fe(OH)<sub>3</sub> and Mg(OH)<sub>2</sub> to eliminate the influences of the sample matrix before analyzing REEs on the ICP-MS [18].

## Materials and Methods

### *Sample Collection and Preservation*

The sixteen samples of soil were collected from four areas in Vietnam (Fig. 1). Tea plantation soils were collected in the Northwest (i.e., Lai Chau and Yen Bai provinces are mainly characterized by gray and red-yellow soils), Northeast (i.e., Ha Giang, Phu Tho, and Thai Nguyen provinces are mainly presented by gray soil, red-yellow, feral soil on claystone and hilly soil, respectively). In the Central Highlands, soil samples for tea and coffee cultivations were collected in Lam Dong and Dak Lak provinces which are characterized by mainly lateritic soils on basalt base accounts for a large proportion. In Southeast Vietnam, the rubber plantation soils were taken in Dong Nai, Binh Duong, and Binh Phuoc provinces which are mainly formed on basalt i.e., red and black soils, and ancient alluvium i.e., gray soil, gray-brown soil, yellow-brown soil. The

details of sampling sites are shown in Table 1.

Table 1. Sampling areas and sites.

S.No.	Sampling area	Quantity of Sample	Province	Coordinates of sampling site
1	Northeast	6	Phu Tho	21°07'59.0"N 105°03'42.5"E
			Thai Nguyen	21°37'54.1"N 105°32'20.5"E
				21°32'39.1"N 105°45'31.5"E
			Ha Giang	22°44'41.8"N 104°54'17.4"E
				22°32'45.1"N 104°47'00.5"E
				22°36'46.1"N 104°45'36.5"E
2	Northwest	5	Yen Bai	21°36'35.7"N 104°35'56.4"E
			Lai Chau	22°26'18.7"N 103°15'55.9"E
				22°15'55.2"N 103°41'41.7"E
				22°17'16.5"N 103°40'49.9"E
			22°25'08.6"N 103°15'27.1"E	
3	Central Highlands	2	Lam Dong	11°36'38.9"N 107°44'13.1"E
			Dak Lak	12°40'32.9"N 108°06'51.2"E
			Dong Nai	10°48'27.5"N 106°56'45.8"E
4	Southeast	3	Binh Duong	11°05'54.4"N 106°37'08.5"E
			Binh Phuoc	11°29'06.7"N 106°34'57.5"E

Soil cores of 30 cm depth were collected in triplicate at each sampling site from the top to the foot of the hill. Samples

were kept cool and transferred to the laboratory where the samples were stored at -18 °C. They were freeze-dried for 48 h, ground and sieved through a 100 µm membrane before analysis.

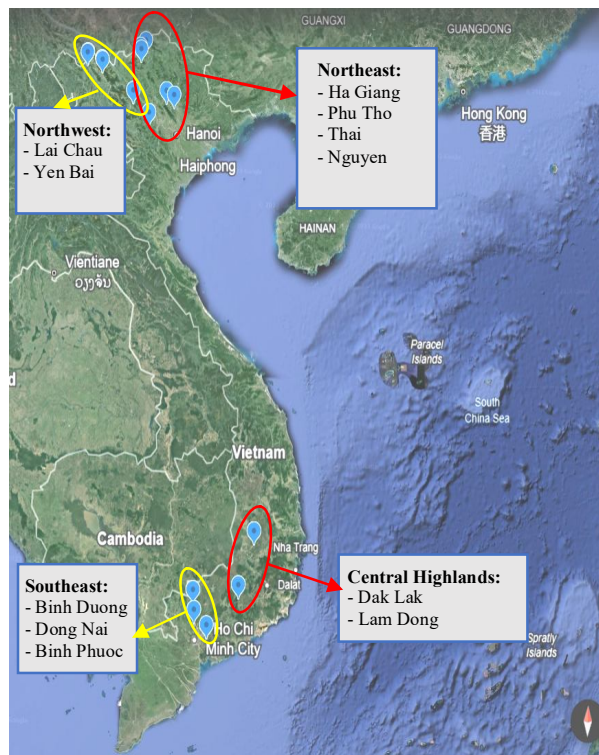


Figure 1. Map of Vietnam, with the circles representing the location of study areas and sampling sites.

### Sample Analysis

**Chemicals and reagents:** lithium tetraborates  $\text{Li}_2\text{B}_4\text{O}_7$ , nitric acid ( $\text{HNO}_3$ ), hydrochloride acid ( $\text{HCl}$ ), Iron(III) nitrate nonahydrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ), magnesium nitrate hexahydrate ( $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ), ammonia solution ( $\text{NH}_4\text{OH}$ ) were purchased from Merck, Germany. The REEs were analyzed by the Agilent ICP-MS 7700, with Kinetic Energy Discrimination, using spiked  $^{197}\text{Au}$  ( $10 \text{ ng mL}^{-1}$ ) as the internal standard (Table 2). The main operation parameters were as follows: RF power ( $1550 \text{ W}$ ), carrier gas ( $1.05 \text{ L min}^{-1}$ ), nebulizer pump ( $0.50 \text{ rps}$ ), and He gas ( $5.0 \text{ L min}^{-1}$ ).

**Table 2.** Limit of detection and quantification m/z of Rare earth elements.

Elements	Quantification m/z	Internal standard	MDL (ng mL <sup>-1</sup> )
Sc	45		0.0953
Y	89		0.0618
La	139		0.1877
Ce	140		0.0881
Pr	141		0.0847
Nd	146		0.0638
Sm	147	<sup>197</sup> Au	0.0652
Eu	153		0.0713
Gd	157		0.0499
Tb	159		0.0328
Dy	163		0.0446
Er	166		0.0394
Yb	172		0.0405
Lu	175		0.0153

### *The Total Content of REEs*

0.1 g ( $\pm 0.0001$ ) of the soil sample and 1.0 g Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> were put into a Pt crucible. Samples were fused at 1000 °C for 30 min. They were dissolved by HCl 3 M and adjusted to 50 mL using distilled water, then centrifuged to reject residual pellets (Solution A) which were performed co-precipitation with Fe(OH)<sub>3</sub> and Mn(OH)<sub>2</sub>, including two steps prior to analyzing REEs on ICP-MS [35].

Step I: 10 mL of Solution A was transferred into the plastic tube and added 2.5 mL Fe<sup>3+</sup>:Mg<sup>2+</sup> (5000:5000 mg L<sup>-1</sup>), which was then adjusted to 25 mL using distilled water. The mixtures of 2.0 mL triethanolamine 50% (v/v) and 6.0 mL NaOH 30% (m/v) were added into the tube and waited for 15 min. The sample was then centrifuged to collect the solid which was dissolved using 0.5 mL concentrated HNO<sub>3</sub> (Solution B).

Step II: 10 mL of distilled water was added into solution B and NH<sub>4</sub>OH 50% (v/v) was then used to adjust the pH to unstill appearing iron precipitation. 10 mL of buffer pH = 9 was added in and kept for 15 min, and centrifuged to collect the solid which was

cleaned using 30 mL of distilled water and 0.5 mL of concentrated NH<sub>4</sub>OH was added and then centrifuged to collect the solid. The solid phase was dissolved using 0.5 mL concentrated HNO<sub>3</sub> and adjusted to 25 mL of distilled water (Solution C) which was analyzed for REEs contents on ICP – MS.

### *The Exchangeable Content of REEs*

2.0 g of soil sample was weighed into a 50 mL plastic tube and added 30 mL of ammonium acetate 1 M (pH = 4). The sample was shaken for 45 min at 300 rpm and centrifuged to reject residual pellets which was adjusted to 50 mL using distilled water. The content of REEs was analyzed on ICP – MS.

### *Statistical Analysis*

The REEs content was calculated based on the standard curve method. For samples taken according to altitude, we use the ANOVA statistical standard, based on the p-value to see if there is a difference in REEs content when altitude changes.

## **Results and Discussion**

### *Assessment of Mean REEs Content*

The mean content of REEs was 238 mg kg<sup>-1</sup>. The content of LREEs was higher than the HREEs (accounting for 80.8% of the total amount and 7.5 fold higher than that of the HREEs) (Table 3). A similar variability was also found in some countries such as Australia, Malaysia, the USA, and China (Table 4). The results also showed that the content of REEs in the soil follows the Oddo Harkins rule: "Elements with even atomic numbers will have a greater concentration than elements with adjacent odd-numbered atoms", for example, Ce (Z = 58) has a higher content than La (Z = 57) and Pr (Z = 59), similarly Yb (Z = 70) has a higher content

than Lu ( $Z = 71$ ). The content of REEs follows the propensity:  $Sc > Y < La < Ce > Pr < Nd > Sm > Eu < Gd > Tb < Dy > Er > Yb > Lu$  is shown in Fig 2. The presence of REEs was according to the accumulated content in the soil. The publications showed that the content of REEs decreased in the order  $Ce > La > Nd > Pr > Sm, Gd > Dy > Er, Yb > Eu$  (Table 4). However, in the present study, the content of REEs was as follows  $Ce > Nd > La > Pr > Sm, Eu > Yb$ . These differences may come from soil characteristics according to geographical areas and farming conditions [3, 6].

Table 3. The mean content of REEs from four areas in Vietnam.

Elements	Content range (mgkg <sup>-1</sup> )	Mean content (mgkg <sup>-1</sup> )
Sc	3.52 – 66.78	20 ± 17
Y	2.92 – 40.17	15 ± 10
La	12.11 – 75.35	36 ± 22
Ce	13.82 – 169.00	80 ± 52
Pr	2.43 – 27.95	12.4 ± 9.2
Nd	6.86 – 107.28	45 ± 33
Sm	0.93 – 21.34	9.1 ± 6.7
Eu	0.15 – 5.62	1.8 ± 1.7
Gd	0.73 – 14.45	7.7 ± 5.6
Tb	0.17 – 4.23	1.4 ± 1.2
Dy	0.39 – 12.65	5.0 ± 3.8
Er	0.24 – 5.88	2.1 ± 1.6
Yb	0.13 – 4.37	1.5 ± 1.2
Lu	ND – 1.55	0.21 ± 0.36

ND: no detection

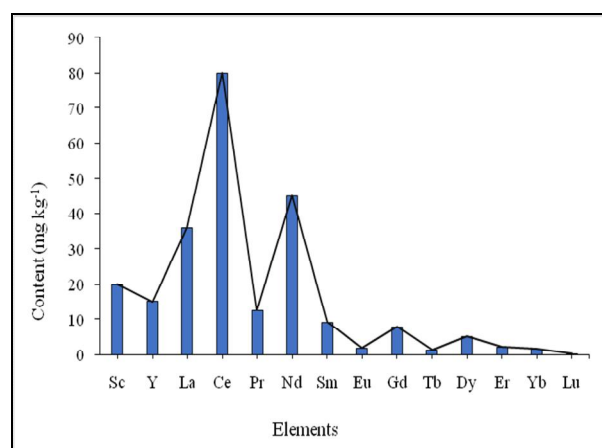


Figure 2. Distribution pattern of REEs content in the present study

The LREEs contents in the present study were higher than the mean values in some parts of the world e.g., Australia, Malaysia, the United States, and China, especially for Ce and Nd (Table 4). We suggest that the difference in REEs content could be related to the investigated samples collected in the northern mountainous areas where the rare earth mines were concentrated such as Dong Pao, Nam Xe, and Yen Phu. The phenomenon was also consistent with the previous publication reported by Li et al. [19]. While, for the HREEs, except for Tb and Dy, the other elements had lower contents than those measured in the Earth's crust and lower than in Malaysia and China.

Table 4. Content of REEs (mgkg<sup>-1</sup>) in some countries in the world [5, 20].

Elements	Australia	Malaysia	USA	China	Jiangxi Mine (China)	Baotou Mine (China)
Sc	-	-	-	-	-	-
Y	-	-	-	-	51.23	-
La	15.38	30.46	13.62	37.57	208.5	55.03
Ce	60.49	52.75	25.67	77.32	351.63	132.6
Pr	4.13	-	2.45	7.87	46.9	12.02
Nd	14.63	28.75	9.98	29.31	159.75	42.93
Sm	2.76	4.88	1.4	5.75	24.98	6.7
Eu	0.64	0.96	0.37	1.17	2.36	1.25
Gd	2.58	21.13	2.82	5.	21.68	5.43
Tb	0.43	1.3	0.14	0.81	2.54	0.76
Dy	2.06	4.98	0.7	4.66	11.09	4.37
Er	0.79	5.53	<0.003	2.68	5.47	2.54
Yb	0.57	2.89	<0.008	2.55	4.58	2.53
Lu	0.08	0.88	<0.01	0.42	0.68	0.40

(-) No data available

### The Geographical Distribution of REEs

The contents of REEs in the four areas are shown in Table 5. The highest

content of REEs was measured in the North West i.e., with a mean value of  $357.7 \text{ mg kg}^{-1}$ , following the Northeast ( $283.9 \text{ mg kg}^{-1}$ ), Central Highlands ( $182.4 \text{ mg kg}^{-1}$ ), and the lowest contents in the Southeast ( $89.6 \text{ mg kg}^{-1}$ ) that because the geological structure is mainly gray soils with poor mineral resources [3]. Other areas e.g., the Central Highlands and the North of Vietnam with the terrain being midland and mountain, the diversity of soil types and also the content of minerals. The total content of REEs in those two areas was quite high content to those measured in other ones that may result from the cultivation of perennial crops such as tea, coffee, rubber, etc., so a high amount of REEs also dominated the soil through fertilizers during cultivation [1].

**Table 5.** Contents of REEs in the Southeast, Central Highlands, Northwest, and Northeast of Vietnam.

Parameter	Southeast	Central Highlands	Northwest	Northeast
Mean content ( $\text{mg kg}^{-1}$ )	89.6	182.4	357.7	283.9
LREEs ( $\text{mg kg}^{-1}$ )	72.0	117.3	284.	225.7
HREEs ( $\text{mg kg}^{-1}$ )	10.5	29.8	43.2	43.6
$\frac{\text{LREEs}}{\text{HREEs}}$	6.9	3.9	6.6	5.2
$\frac{\text{LREEs}}{\text{REEs}}$	80.3	64.3	79.4	79.5

In addition, the content of REEs in the Northern was higher than that in the Central Highlands and the Southeast and was higher than those measured in the Earth's crust due to the dependence on geographical location and characteristics. The soil in the Northern area is formed from the weathering of rocks such as granite, gneiss, and metamorphic [1] containing minerals of monazite, xenotime, fluor carbonates, and bastnasite. In particular, two rare earth mines at Nam Xe (Phong Tho) and Dong Pao (Tam Duong) are known in Lai

Chau province and the Yen Phu mine is known in Yen Bai province which could increase REEs in the adjacent soil areas. The results are consistent with previous publications on REEs in soils close to rare earth mines in China (Table 4) [20]. The ratio of LREEs/REEs in all areas was high (over 60%) and the ratio of LREEs/HREEs in these areas exhibited a rather large disparity, e.g., in the Southeast (6.9), while the Central Highlands (3.9), the Northwest (6.6) and the Northeast (5.2). The result was similar to the previous publication with the proportion of LREEs accounting for 5.04 – 9.06 times which has been reported by Lian et al. [21]. These results show that the elements of the LREEs group dominated the total content in all study areas with the proportions of 80.3% in the Southeast, 79.4% in the Northeast, and 79.5% in the Northwest, which are much higher 64.3% compared to the Central Highlands. The reason for the high percentage of LREEs is that they often exist in many minerals such as monazite (Ce, La, Nd, Sm), carbonates and fluor carbonates (La), bastnasite (La, Nd), while HREEs exist mainly in xenotime minerals (Y, Yb) with low reserves and unconcentrated distribution.

In Lai Chau, Ha Giang, and Thai Nguyen provinces, samples were collected according to altitude so we used the ANOVA statistical standard to evaluate the difference in REEs content by using a probability value (p) (Table 6). There was a significant difference in the contents of all REEs by altitude in Lai Châu ( $p < 0.05$ ). However, in Thai Nguyen and Ha Giang provinces, only some elements presented the least variation by altitude ( $p > 0.05$  for La, Pr, Nd, Sm, Gd, Tb). In general, the contents of REEs were mostly concentrated in low terrain and this distribution was also highly dependent on the geographical location of each. The distribution

of REEs is uneven between areas (Table 5) and is influenced by several factors. In each area, the REEs content also fluctuates depending on altitude. Factors, i.e., the effects of weathering processes, erosion, and landslides cause sedimentation and increase REEs content in low-altitude locations [21,22]. In addition, the effects of farming activities, using fertilizers supplemented with REEs [23] and the processes of mining and transporting minerals and ores containing REEs also contribute to an increase in the REEs content in the soil.

**Table 6.** A probability value (p) of the elements concerning altitude using ANOVA analysis.

Elements	Lai Chau	Ha Giang	Thai Nguyen
Sc	$3.75 \times 10^{-6}$	$1.29 \times 10^{-3}$	$3.17 \times 10^{-4}$
Y	$1. \times 10^{-5}$	$1.35 \times 10^{-3}$	$6. \times 10^{-3}$
La	$6.77 \times 10^{-4}$	$5.59 \times 10^{-2}$	$3.87 \times 10^{-3}$
Ce	$1.28 \times 10^{-2}$	$8.82 \times 10^{-3}$	$8.32 \times 10^{-4}$
Pr	$2.72 \times 10^{-3}$	$5.31 \times 10^{-2}$	$8.01 \times 10^{-4}$
Nd	$1.22 \times 10^{-3}$	$7.75 \times 10^{-2}$	$1.88 \times 10^{-3}$
Sm	$8.42 \times 10^{-4}$	$4.88 \times 10^{-1}$	$3.03 \times 10^{-3}$
Eu	$5.88 \times 10^{-5}$	$1.58 \times 10^{-1}$	$4.61 \times 10^{-5}$
Gd	$6.02 \times 10^{-4}$	$1.48 \times 10^{-1}$	$1.45 \times 10^{-3}$
Tb	$7.91 \times 10^{-4}$	$5.70 \times 10^{-2}$	$5.73 \times 10^{-3}$
Dy	$6. \times 10^{-5}$	$1.33 \times 10^{-2}$	$5.39 \times 10^{-2}$
Er	$2.15 \times 10^{-5}$	$1.74 \times 10^{-3}$	$9.52 \times 10^{-3}$
Yb	$1.66 \times 10^{-5}$	$6.25 \times 10^{-4}$	$4.40 \times 10^{-5}$
Lu	$1.35 \times 10^{-4}$	$1.84 \times 10^{-3}$	$2.96 \times 10^{-3}$

### Variation of Exchangeable REEs Content

The mean exchangeable content was  $12.7 \text{ mg kg}^{-1}$  in the Southeast,  $23.3 \text{ mg kg}^{-1}$  in the Central Highlands,  $42.9 \text{ mg kg}^{-1}$  in the Northwest, and  $30.7 \text{ mg kg}^{-1}$  in the Northeast which were relatively lower than their total content (Table 7). The highest content of

exchangeable REEs was in the Northwest while the lowest one was in the Southeast; this phenomenon was consistent with the total content variation. The LREEs were still prevailing, the proportions were 85.4% in the Southeast, 67.7% in the Central Highlands, 86.2% in the Northwest, and 91.4% in the Northeast. The difference in the content of the exchangeable and total forms of REEs could be attributed to these elements being tightly bound in the crystal lattice of minerals. The distribution of exchangeable content was quite close to the total content, it depends mainly on topography, climate, and influences from other factors such as farming conditions of human activities, i.e., the main exchangeable was supplied through fertilizers during the cultivation process.

**Table 7.** Variability of exchangeable REEs content.

Parameters	Southeast	Central Highlands	Northwest	Northeast
Mean REEs content ( $\text{mg kg}^{-1}$ )	12.7	23.3	42.9	30.7
Mean LREEs ( $\text{mg kg}^{-1}$ )	10.9	15.8	36.9	28.0
Mean HREEs ( $\text{mg kg}^{-1}$ )	1.63	5.81	4.76	2.27
LREEs/REEs	85.4	67.7	86.2	91.4
LREEs/HREEs	6.7	2.7	7.8	12.3

### Conclusion

The content of REEs ranged from  $0.13$  to  $169 \text{ mg kg}^{-1}$ , of which Ce was the highest content and Lu was the lowest one. They were following  $\text{Sc} > \text{Y} < \text{La} < \text{Ce} > \text{Pr} < \text{Nd} > \text{Sm} > \text{Eu} < \text{Gd} > \text{Tb} < \text{Dy} > \text{Er} > \text{Yb} > \text{Lu}$ . The variation of total content and the exchangeable form exhibited a similar tendency. The LREEs always dominated in soils. The distribution of REEs was not uniform in different areas. In the comparison of four areas in Vietnam including Southeast, Central Highlands, Northwest, and Northeast, it was found that the Northwest had the highest content of REEs, and the Southeast was the lowest one.



We suggest that the distribution of rare earth elements depends on geographical location, climatic conditions, and other factors including farming conditions that increase or decrease the content of these elements in the soil.

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### Conflict of Interest

The authors declare that there is no conflict of interest.

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