



The Mineral Characteristic of Tropical Residual Soil using X-Ray Diffraction (XRD) and Scan Electron Microscopy (SEM)

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(Received: 9th March 2023 ; Revised: 10th April 2023 ; Accepted: 17th May 2023)

Abstract: Volcanic residual soil has unique characteristics due to weather and morphological influences. High temperatures and large amounts of rainfall made the rock weathering intense. Landslide events in volcanic residual soil of West Java are quite serious, more than 100 incidents in total a year. Landslides that occur due to unsaturated conditions on the top of the slope become saturated, so it is very important to conduct research on these tropical residual soil weathering zones to define its mineral characterization using Scan Electron Microscopy (SEM) and X-Ray Diffraction (XRD). From the research it was found that the dominant mineral types and contents were Halloysite and Kaolinite minerals. A typical mineral profile is found that is Goethite and Hematite minerals at the soil surface, Carbonate and Chlorite minerals in deeper soils and large amounts of Feldspar minerals in more deep layers. Because there are unique minerals at each depth of soil weathering, zone classification can be done easily starting from the Initial Leaching Zone at the deepest part where there is Feldspar, the Mineral Washing Zone there is Carbonate and Chlorite and the Oxidation Zone on the surface (there are Oxide and Hydroxide minerals). So that it can form a Residual Tropical Volcanic Weathering Profile.

Keywords: *Initial Leaching Zone; Mineral Washing Zone; Oxidation Zone*

1. Introduction

Residual soil is formed due to weathering of rocks and is above the original rock, while tropical soil is soil that lies between the Tropic of Cancer (23.5°) in the North and the Tropic of Capricorn (23.5°) in the South. The stage of weathering from the existing rock to the residual soil is an independent variable that determines the other residual soil characteristics. Landslides in West Java occur quite frequently, more than 100 incidents per year. Most of landslides are caused by a heavy rainfall where the shear strength at the top of the slope surface reduced as the surface zone become saturated from unsaturated stage. So that at the beginning of the research it is necessary to determine the composition of the residual soil material to determine the characteristics of the soil weathering stages [1]. The following is a Geological map of Sheet Cianjur, West Java.

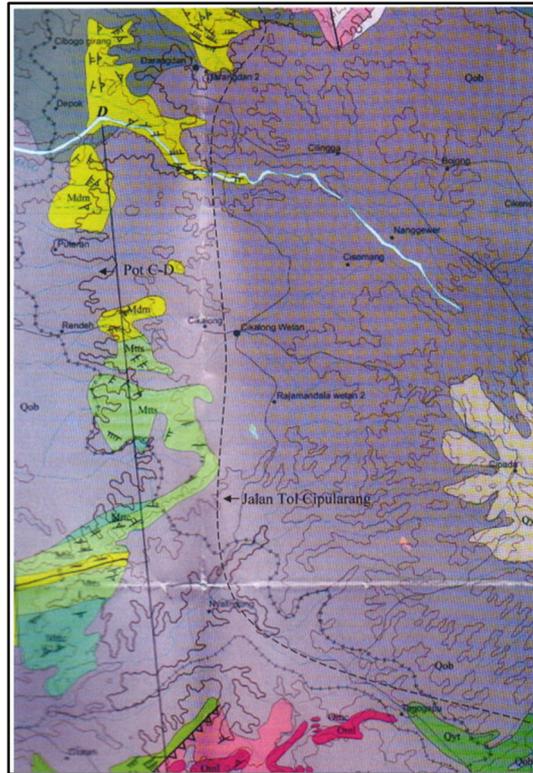


Fig. 1. Sheet Geology Map of Cianjur [2] where the formation describe as Qob, Old Volcanic Soil.

Wesley (1973) on residual tropical volcanic soils of West Java, concluded that the soil has specific characteristics in which high shear strength parameters are obtained, even though the soil has a large clay mineral content but it is included in the specification of soil with low activity [3]. Tuncer and Lohnes (1977) published a parameter profile of mineral content, physical properties and shear strength of weathered residual soils based on a research of residual soils in Hawaii and Puerto Rico. This weathering profile is very interesting because it shows very different conditions in each weathering zone. By compiling a profile of the same parameters, the deeper knowledge will be obtained regarding the mechanism of the two factors, structure and suction matrix in controlling shear strength on slopes [4].

Soil properties depend on two main factors, namely composition and structure. The composition includes the properties of the grains themselves, namely their size, shape and type of mineralogy. The composition of tropical residual soils, high temperatures and large amounts of rainfall make rock weathering intensive. Weathering characteristics of the rock consist of Feldspar and ferromagnesian which are fragmented, Silica and Bases (Na_2O , K_2O , CaO , MgO) which are leached (disappear) and iron and aluminium oxides which are concentrated.

2. Results of Residual Soil Mineral Structure by XRD method

In determining the dominant type of mineral, an X-Ray Diffraction test is performed. With this test it can be determined what minerals are found in the soil sample. In the test, a relatively small sample volume was taken in the test. The minerals expected to be found in the residual soil are montmorillonite, kaolinite and gibbsite minerals [5].

The measurement result obtained is the amount of intensity per step (time step) by carrying out a time series statistical analysis. The measurement results per step are converted into a graph of

intensity peaks which is also called a diffractogram. The following is the result of a sample diffractogram.

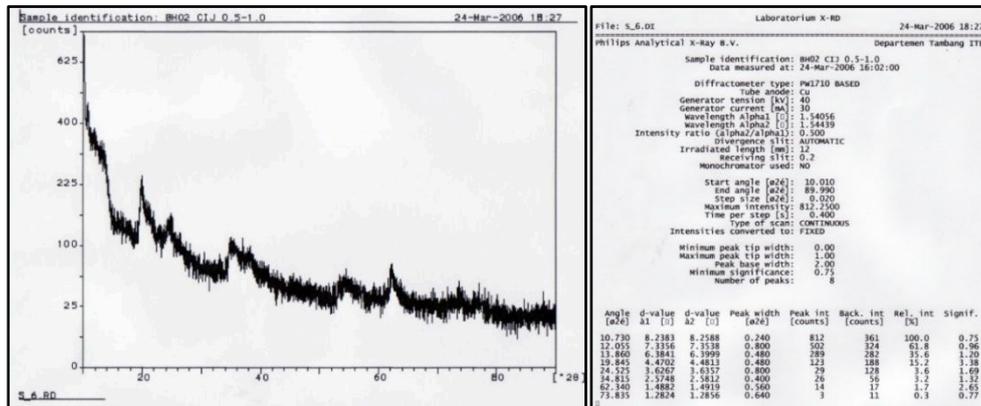


Fig. 2. Diffractogram sample results.

Peak width is the width of a peak on the diffraction diagram. Peak intensity is the magnitude of the intensity of a peak (counts). Background intensity is the intensity of the background in a sample. Relative intensity is the intensity of a peak to the highest peak measured in the sample.

The results of the tests were obtained at a depth of 0.50-1.00. The minerals found were Goethite/Hematite, Halloysite and Kaolinite. Goethite and Hematite minerals are produced from the oxidation process of iron minerals. Halloysite and Kaolinite are minerals which are groups of silica tetrahedral layers connected to octahedral aluminium layers.

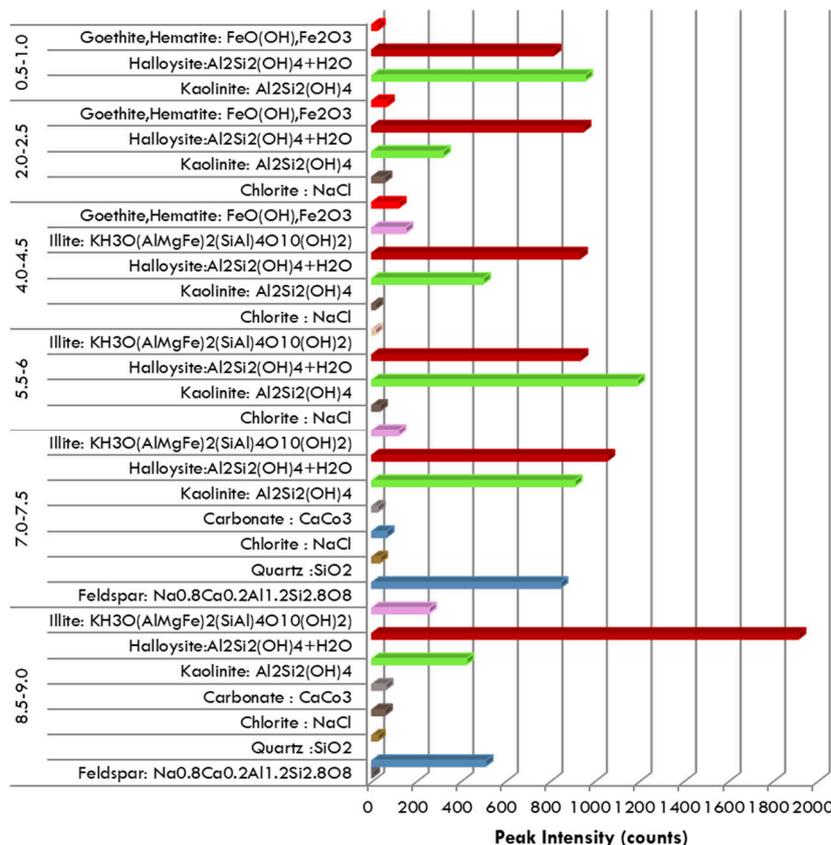


Fig. 3. Mineral Profile at BH02 Cijengkol Slope.

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At a depth of 2.00-2.50 m the minerals found are Goethite/Hematite, Halloysite, Kaolinite and Chlorite. Chlorite is a mineral that has a large pH (potential hydrogen), which is alkaline. At a depth of 4.00 – 4.50 m the minerals found are Goethite/Hematite, Illite, Halloysite, Kaolinite and Chlorite. At a depth of 5.50 – 6.00 minerals found are Illite, Halloysite, Kaolinite and Chlorite. The presence of Chlorite in the 2.00 to 6.00 m layer indicates that this depth zone is a zone of alkaline mineral leaching. At depths of 7.00-7.50 minerals found are Illite, Halloysite, Kaolinite Carbonate, Chlorite, Quartz and Feldspar. At a depth of 8.50-9.00 m the materials found are Illite, Halloysite, Kaolinite, Carbonate, Chlorite, Quartz and Feldspar. The composition of these minerals can be seen in the following Figure 3.

If we only consider outside Silicate Group, except Feldspar we have the composition as follow:

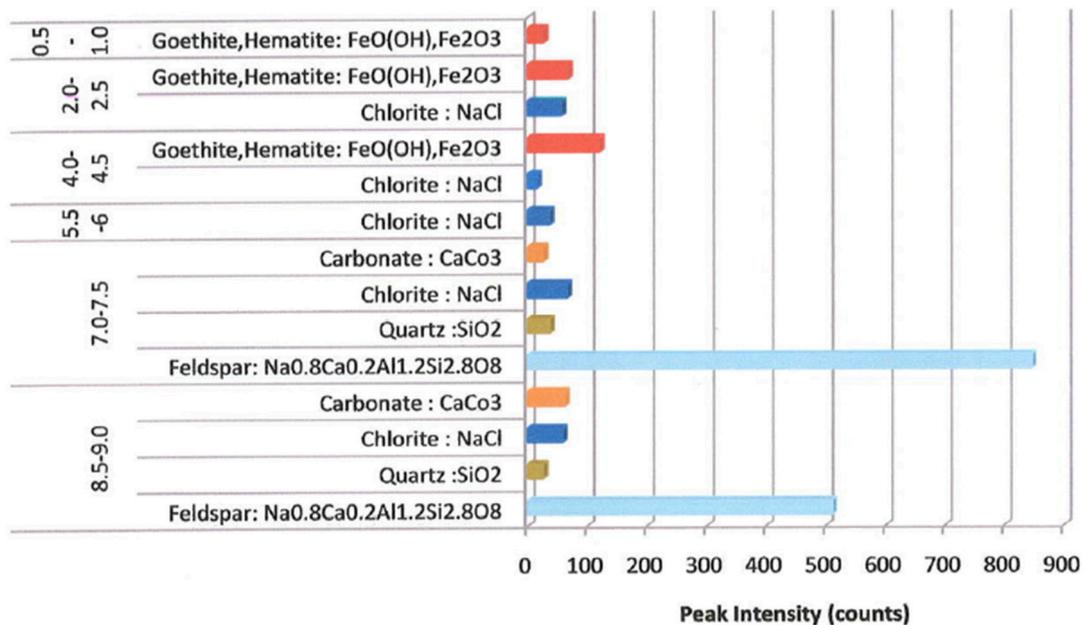


Fig. 4. Mineral Profile at BH02 Cijengkol Slope outside Silicate Group except Feldspar.

Goethite and Hematite are found in layers 0.5-1.0 to 4.0-4.5 m, this indicates an oxidation process of iron minerals which are not easily dissolved by water and then oxidized by air. From the mineral profile above several different zones as follows:

- At a depth of 7.0 – 7.5 m to 8.5 – 9.0 m is the initial zone of weathering because large amounts of feldspar minerals are still found.
- At a depth of 2.0 – 2.5 m to 5.5 – 6.0 is a leaching zone of alkaline minerals (alkaline), namely carbonate and chlorite, where amount of carbonate and chlorite is less than in the initial zone of weathering below.
- At a depth of 0.5 – 1.0 m to 4.0 – 4.5 m is an oxidation zone because there are minerals from the oxidation of Goethite and Hematite.

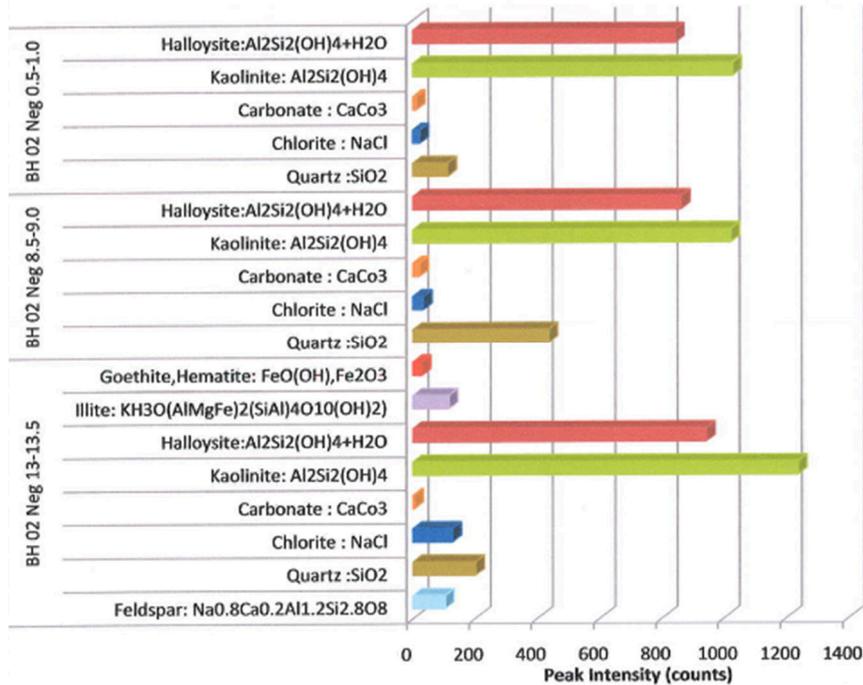


Fig. 5. Mineral Profile at BH02 Neglajaya slopes.

Based on the mineral profile at BH02, the Neglajaya slope (slope to the left from Jakarta), the mineral content is as follows:

- At a depth of 13.0 – 13.5 m, the minerals Goethite/Hematite, Illite, Halloysite, Kaolinite Carbonate, Chlorite, Quartz and Feldspar are found. The presence of the Goethite and Hematite at this depth indicating that at a certain time this layer is at the oxidation zone. The presence of Feldspar minerals were found but the presentation is far below the Kaolinites, that indicates that the layer is not the initial weathering zone.
- At a depth of 8.50 – 9.0 m, the minerals Halloysite, Kaolinite, Carbonate, Chlorite and Quartz are found. The presence of these minerals indicates that the layer is in the alkaline mineral washing zone. The amount of quartz content determines the type of soil that is silt (silt clay).
- At a depth of 0.5 – 1.0 m, the minerals Halloysite, Kaolinite, Carbonate, Chlorite and Quartz are found. The missing of Goethite and Hematite at this layer suggest that this layer is not the oxidation zone.

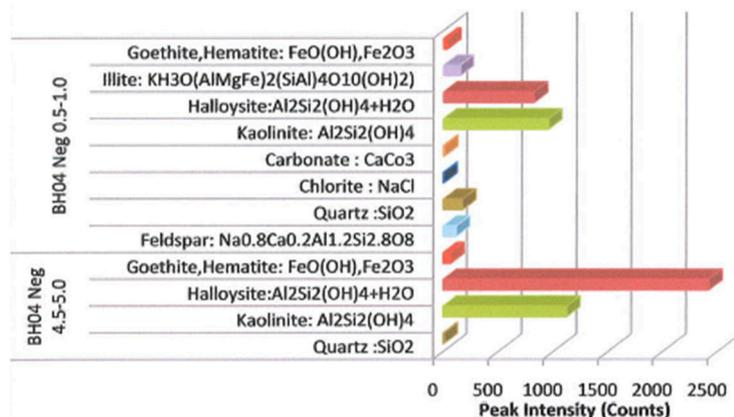


Fig. 6. Mineral Profile at BH04 Neglajaya slope (slope to the left from Jakarta).

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Based on the BH04 profile of the Neglajaya slope (slope to the right from Jakarta) there are Carbonate, Chlorite and a little Feldspar at a depth of 0.50-1.00 m and not found at a depth of 4.00-5.00 m indicating that the piled up or colluvial soil has been transported.

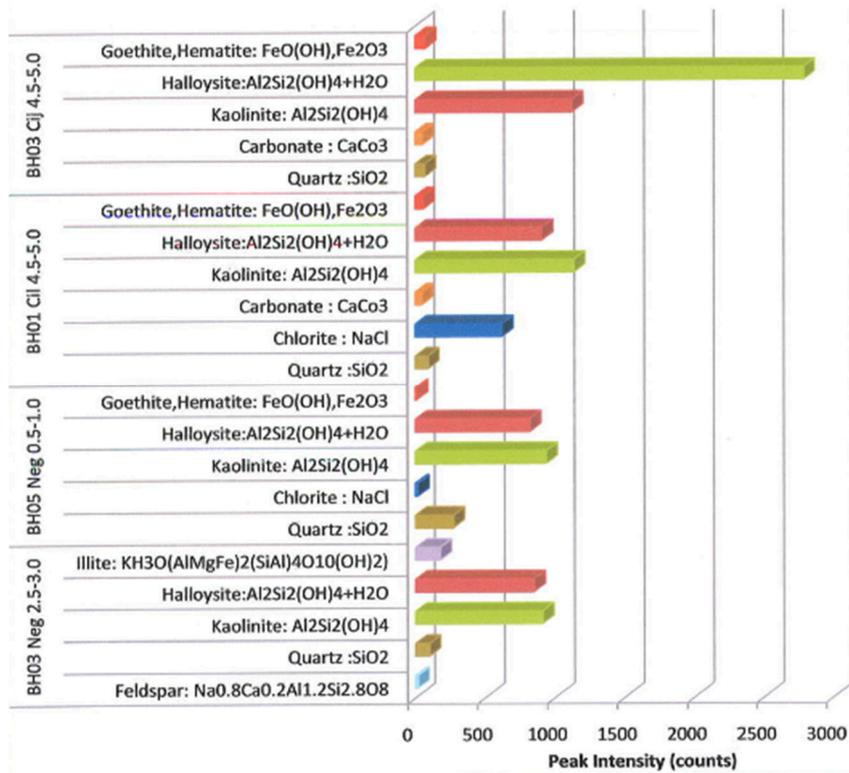


Fig. 7. Mineral Profiles BH03 Cijengkol, BH01 Cilame, BH03 Neglajaya and BH05 Neglajaya.

Mineral content found in various layers on different soil profiles, namely the slopes of Cijengkol BH03 4.5 – 5.0 m deep, the slopes of Cilame BH01 4.5 - 5.0 m deep, the right slope of Neglajaya BH05 0.5 – 1.0 m deep, the left slope of Neglajaya BH03 2.5 – 3.0 m deep. There are similarities in the minerals found in BH03 at depths of 4.5-5.0 m and BH01 at depths of 4.5-5.0 m, both of which are in the alkaline mineral washing zone. In the 0.5 – 1.0 m layer on BH05 the right slope of Neglajaya there are mineral similarities with layers 0.50-1.00 BH02 where Chlorite minerals are found. In the 2.50 – 3.00 m layer at BH03 there are Illite, Halloysite, Kaolinite, Quartz and Feldspar minerals. This layer shows the end zone of alkaline mineral washing because there are no Carbonate and Chlorite minerals.

3. Research Result of Residual Soil Mineral Structure with SEM Method

The test of the soil minerals composition using the Scan Electron Microscopy (SEM) method with the results of photomicrograph testing with magnifications of 2500 x and 5000 x there are soil profile samples of BH02 Cijengkol as follows:

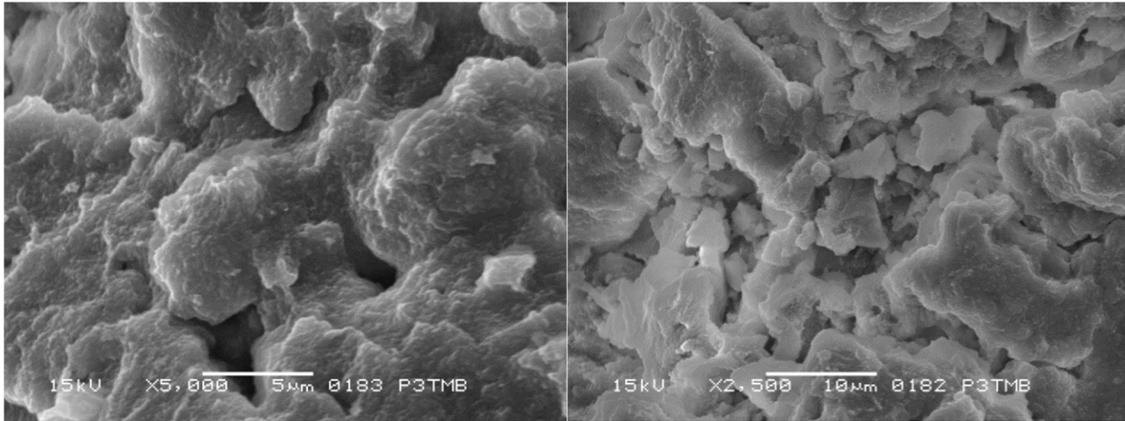


Fig. 8. Cij BH02 0.50 – 1.00 m Magnification of 2500 x and 5000 x

The SEM results on the 0.50-1.00 m layer of soil are shaped like layers of plates stacked up like an accordion. Minerals that are shaped like this are known to be the mineral kaolin measuring 7.5 µm to 15 µm.

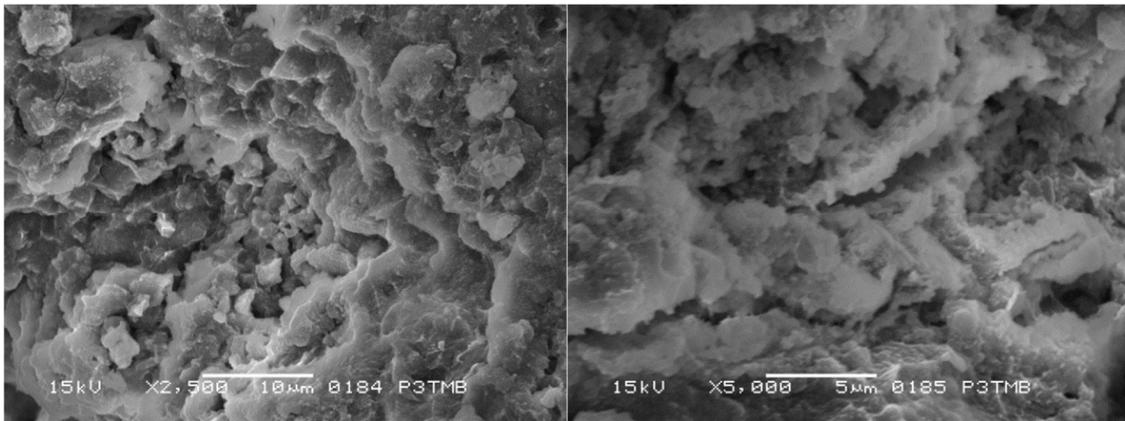


Fig. 9. Cih BH02 2.00 – 2.50 m Magnification of 2500 x and 5000 x

In the SEM photo, the 2.00 – 2.50 m layer of soil can be seen that there are still stacked plate-shaped layers, euhedral (hexagon) kaolin minerals but not perfect. The size of the mineral plate is smaller than the previous layer.

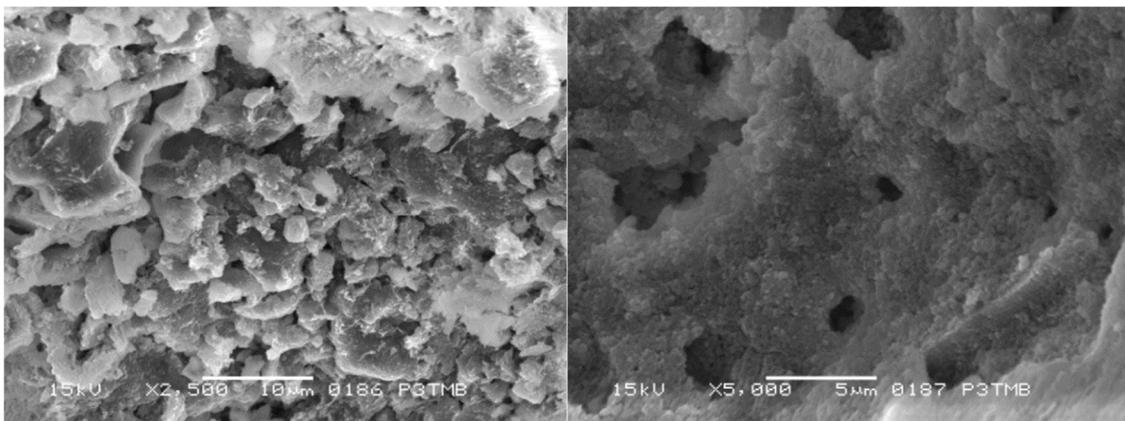


Fig. 10. Cij BH02 4.00 – 4.50 m Magnification of 2500 x and 5000 x

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In this layer, the size of the kaolin mineral plate is the smallest compared to other layers.

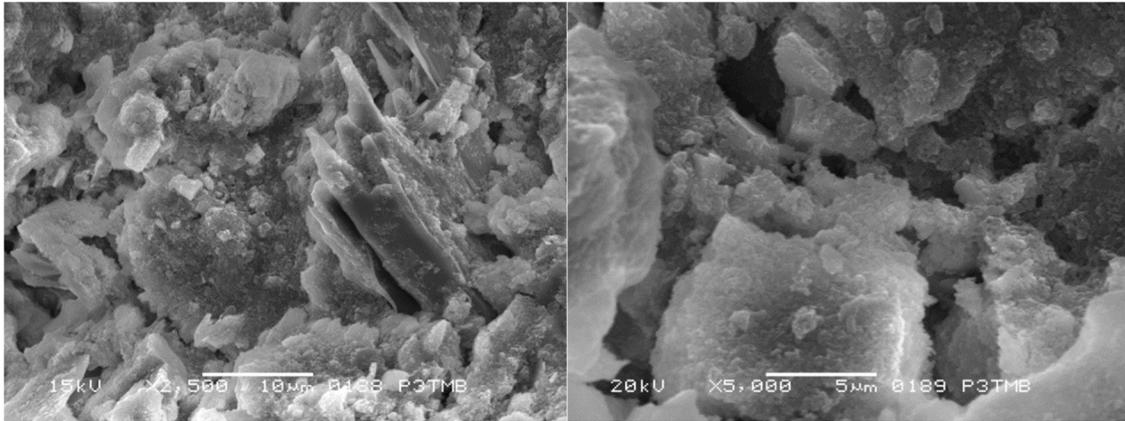


Fig. 11. Cij BH02 7.00 – 7.50 m Magnification of 2500 x and 5000 x

In the 7.00 - 7.50 m layer, it can be seen that most of the minerals are still in the form of plates (kaolin minerals) however, crystal forms are already visible which indicate the presence of original block-shaped minerals (feldspar).

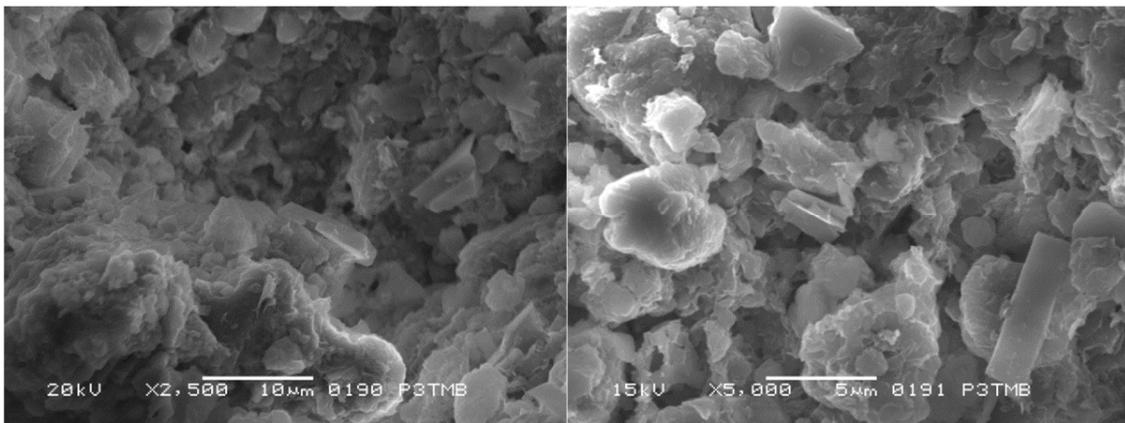


Fig. 12. Cij BH02 8.50 – 9.00 m Magnification of 2500 x and 5000 x

In layers of 8.50 – 9.00 m it appears that block mineral forms are more visible and scattered compared to layers of 7.00 – 7.50 m. Testing with Energy Dispersive X-Ray was carried out to obtain the dominant elements found in each soil layer. The results obtained are X-Ray graphs with energy units as follows.

Table 1. The types of minerals found in the soil layer profile of Cijengkol BH02

BHO2 0.50 - 1.00 m		BHO2 2.00 - 2.50 m		BHO2 4.00 - 4.50 m		BHO2 8.50 - 9.00 m	
Element	avg mass (%)						
O	48.13	O	45.39	O	46.99	Fe K	32.67
Si K	25.04	Si K	22.90	Si K	24.55	O	32.64
Al K	18.51	Al K	18.09	Al K	18.44	Si K	19.01
Fe K	13.19	Fe K	13.34	C K	9.61	C K	16.70
Mo L	11.35	C K	10.80	Zr L	9.37	Zr L	6.09
Zr L	8.77	Ba L	2.93	Fe K	5.87	U M	4.69
		P K	0.56	Se L	1.46	Ca K	3.88
		S K	0.20	Ti K	0.79	Pr L	2.39
		Sn L	0.02	P K	0.74	Pd L	2.20
				Tl M	0.52	Tl M	2.00
						Al K	1.71
						Mn K	1.47
						Ru L	1.28
						V K	0.97
						Cd L	0.89
						Cr K	0.70
						S K	0.56
						Ti K	0.25
						Sb L	0.17

Based on table above, the minerals contained in each layer of soil are as follows:

- Mineral O is the mineral with the highest average mass percentage in each layer except in the 8.50 – 9.00 m layer where the highest average mass percentage is Fe minerals
- Mineral Si is a mineral that is not easily dissolved (leached) has a consistent average mass percentage in each layer, as well as Zr minerals
- Mineral Ca²⁺ which is the most easily leached mineral after Na⁺, K⁺, Mg²⁺ appears to be remaining at a depth of 8.50 – 9.00 m
- Mineral Al³⁺ from 1.71% in the soil layer 8.50 – 9.00 m increased to approximately 18% at a depth of 4.00 – 4.50 m to a depth of 0.50 – 1.00 m
- Mineral Fe from the large rock elements (such as ferrohornblende from granite) at a depth of 8.50 – 9.00 m decreases at a depth of 4.00 – 4.50 m then increases again at a depth of 2.00 – 2.50 m to 0.50 – 1.00 m due to the oxidation process
- Mineral C as a weathering agent from rainwater content appears to have the largest mass percentage at a depth of 8.50 – 9.00 m indicating that the weathering process has started at this considerable depth.

4. Results of Residual Soil Mineral Structures based on comparison of XRD and SEM

4. 1 Weathering/laterization processes

The comparison found with the Energy Dispersive X-Ray method from the SEM tool shows in outline the laterization characteristics where the addition of iron oxide minerals occurs. It is

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confirmed that the results of the mineral composition obtained by the X-Ray method as shown in the following comparison tables and figures.

Table 2. Comparison Al₂O₃ to SiO₂ and the ratio FeO to SiO₂ in each soil layer on the slopes of Cijengkol

Depth	Mass Comparisson	
0.5 - 1.0 m	Al ₂ O ₃ /SiO ₂	0.599
	FeO/SiO ₂	0.277
	(FeO + Al ₂ O ₃) / SiO ₂	0.668
2.0 - 2.5 m	Al ₂ O ₃ /SiO ₂	0.711
	FeO/SiO ₂	0.366
	(FeO + Al ₂ O ₃) / SiO ₂	0.985
4.0 - 4.5 m	Al ₂ O ₃ /SiO ₂	0.664
	FeO/SiO ₂	0.149
	(FeO + Al ₂ O ₃) / SiO ₂	0.775
8.5 - 9.0 m	Al ₂ O ₃ /SiO ₂	0.116
	FeO/SiO ₂	1.637
	(FeO + Al ₂ O ₃) / SiO ₂	1.714

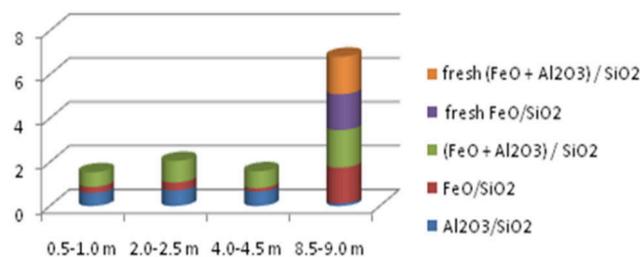


Fig. 13. Comparison of Al₂O₃ to SiO₂ and the ratio of FeO to SiO₂ in each soil layer on the slopes of Cijengkol

4.2 Early Weathering Zone

Based on the following table, the large FeO/Al₂O₃ ratio is found at a depth of 8.5 – 9.0 then it is greatly reduced at a depth of 4.0 – 4.5 m, and then the percentage increases slightly towards the surface. The value of FeO/Al₂O₃ and decreasing percentage indicates that the original rock is mafic or ultramafic rock, which in accordance with the reality in the resulting laterite field is a fertile residual soil.

Table 3. Comparison of Al₂O₃ to SiO₂, FeO to SiO₂ dan FeO to Al₂O₃ in each soil layer on the slopes of Cijengkol

Depth	Mass Comparisson	
0.5 - 1.0 m	Al ₂ O ₃ /SiO ₂	0.599
	FeO/SiO ₂	0.277
	(FeO + Al ₂ O ₃) / SiO ₂	0.437
2.0 - 2.5 m	Al ₂ O ₃ /SiO ₂	0.711
	FeO/SiO ₂	0.366
	(FeO + Al ₂ O ₃) / SiO ₂	0.388

Depth	Mass Comparisson	
4.0 - 4.5 m	Al ₂ O ₃ /SiO ₂	0.664
	FeO/SiO ₂	0.149
	(FeO + Al ₂ O ₃) / SiO ₂	0.228
8.5 - 9.0 m	Al ₂ O ₃ /SiO ₂	0.116
	FeO/SiO ₂	1.637
	(FeO + Al ₂ O ₃) / SiO ₂	12.460

4.3 Alkaline Mineral Leaching Zone

Table 4. Mineral alalin at the depth of 2.0 – 2.5 m up to 8.5 – 9.0 m BH02 Cijengkol

Depth	Element	Count
2.0 - 2.5 m	Chlorite : NaCl	56
4.0 - 4.5 m	Chlorite : NaCl	15
	Illite: KH ₃ O(AlMgFe) ₂ (SiAl) ₄ O ₁₀ (OH) ₂	151
5.5 - 6.0 m	Chlorite : NaCl	36
	Illite: KH ₃ O(AlMgFe) ₂ (SiAl) ₄ O ₁₀ (OH) ₂	14
7.0 - 7.5 m	Chlorite : NaCl	66
	Carbonate : CaCO ₃	26
	Illite: KH ₃ O(AlMgFe) ₂ (SiAl) ₄ O ₁₀ (OH) ₂	119
8.5 - 9.0 m	Chlorite : NaCl	58
	Carbonate : CaCO ₃	61
	Illite: KH ₃ O(AlMgFe) ₂ (SiAl) ₄ O ₁₀ (OH) ₂	256

Table 5. Al₂O₃ and SiO₂ at the depth of 0.5 – 1.0 m up to 4.0 – 4.5 m BH02 Cijengkol

0.5 - 1.0 m			2.0 - 2.5 m			4.0 - 4.5 m		
Compound	Cation	Sum of Cation	Compound	Cation	Sum of Cation	Compound	Cation	Sum of Cation
P1	Al ₂ O ₃	5.75	P1	Al ₂ O ₃	6.37	P1	Al ₂ O ₃	5.44
	SiO ₂	7.69		SiO ₂	5.91		SiO ₂	6.67
P2	Al ₂ O ₃	4.37	P2	Al ₂ O ₃	6.14	P2	Al ₂ O ₃	5.23
	SiO ₂	7.31		SiO ₂	7.2		SiO ₂	7.31
P3	Al ₂ O ₃	5.5	P3	Al ₂ O ₃	4.8	P3	Al ₂ O ₃	5.95
	SiO ₂	5.95		SiO ₂	7.35		SiO ₂	7.31
P4	Al ₂ O ₃	6.22	P4	Al ₂ O ₃	5.42	P4	Al ₂ O ₃	5.7
	SiO ₂	7.33		SiO ₂	7.08		SiO ₂	7.26
Average		12.53			12.57			12.72

Based on the following table, there is a reduction in the number of cations from a depth of 4.0 – 4.5 m to 0.5 – 1.0 m. Reduced cations in the elements Al₂O₃ and SiO₂ can indicate the phenomenon of reduced CEC values in the profile. The same is true for the profile of BH02 Neglajaya where the number of cations decreases from a depth of 13.0 – 13.5 m to a depth of 0.5 – 1.0 m.

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Table 6. Al₂O₃ and SiO₂ at the depth of 0.5 – 1.0 m to 4.0 – 4.5 m BH02 Neglajaya

0.5 - 1.0 m			8.5 - 9.0 m			13.0 - 13.5 m		
Compound	Cation	Sum of Cation	Compound	Cation	Sum of Cation	Compound	Cation	Sum of Cation
P1	Al ₂ O ₃	5.25	P1	Al ₂ O ₃	3.94	P1	Al ₂ O ₃	5.75
	SiO ₂	8.07		SiO ₂	4.12		SiO ₂	7.18
13.31			8.06			12.93		
P2	Al ₂ O ₃	6.61	P2	Al ₂ O ₃	5.55	P2	Al ₂ O ₃	5.62
	SiO ₂	5.80		SiO ₂	7.80		SiO ₂	7.47
12.41			13.35			13.09		
			P3	Al ₂ O ₃	5.25	P3	Al ₂ O ₃	6.27
				SiO ₂	7.05		SiO ₂	7.30
12.87			12.30			13.57		
Average			11.24			13.20		

It can be concluded in general that the alkaline mineral leaching zone is the zone where the highest pH and CEC values are obtained for a residual soil profile. While the leaching zone above is a zone where the pH value and CEC value are relatively smaller.

4.4 Iron Mineral Oxidation Zone (Fe)

From the testing results with XRD that the top layer starting from layers 4.0 – 4.5 m to 0.50 – 1.0 m, is an oxidation zone where the minerals obtained from the oxidation of iron, Hematite and Goethite are obtained. In the following table the amounts of quartz and aluminium minerals are relatively the same, while the amount of iron oxide minerals increasing more than twice from a depth of 4.0 - 4.5 m to a depth of 2.0 - 2.5 m.

Table 7. Mass percentage of Quartz, Silica and Iron Oxide minerals at 4.0 – 4.5 m to 0.5 – 1.0 m

Depth	Mass (%)	
0.5 - 1.0 m	SiO ₂	52.88
	Al ₂ O ₃	31.34
	FeO	16.97
2.0 - 2.5 m	SiO ₂	51.94
	Al ₂ O ₃	34.03
	FeO	17.03
4.0 - 4.5 m	SiO ₂	52.36
	Al ₂ O ₃	34.72
	FeO	7.50

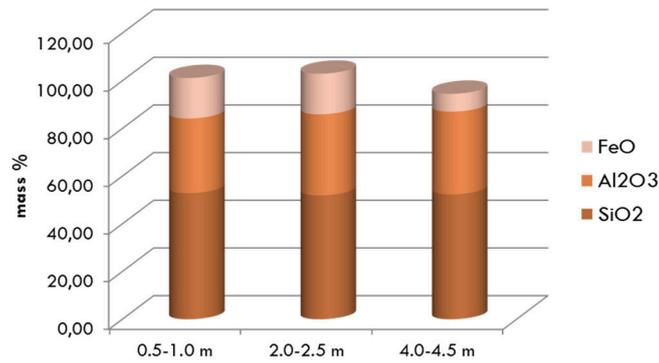


Fig. 14. Mass Percentage of Minerals Quartz, Silica and Iron Oxide at 4.0 – 4.5 m to 0.5 – 1.0 m on the slopes of Cijengkol

The results of the mineral tests on the three samples showed an increase in the percentage of iron oxide and silica to quartz along with the high combustion temperature. This explains that when the soil drying process occurs, the mineral composition of the soil changes, this will affect the physical properties of the soil such as soil plasticity.

Table 8. Comparison of Silica, Quartz and Iron Oxides in native soil samples, 60°C and 100°C oven dry

BH 02 Cijengkol 0.5 - 1.0 m		
Condition:		
Natural Dry	Al_2O_3/SiO_2	0.742
	FeO/SiO_2	0.586
	$(FeO + Al_2O_3) / SiO_2$	1.327
	FeO/Al_2O_3	0.796
60°C oven dry	Al_2O_3/SiO_2	0.721
	FeO/SiO_2	0.209
	$(FeO + Al_2O_3) / SiO_2$	1.615
	FeO/Al_2O_3	0.279
100°C oven dry	Al_2O_3/SiO_2	0.769
	FeO/SiO_2	0.737
	$(FeO + Al_2O_3) / SiO_2$	2.108
	FeO/Al_2O_3	0.880

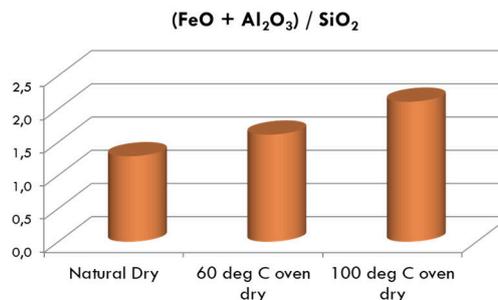


Fig. 15. Percentage of iron oxide and silica to quartz in native soil samples, 60°C and 1100°C oven dry

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From the test results with SEM, there is a significant difference in the size of the kaolin minerals from 1.00 – 5.00 μm at 4.0 – 4.5 m to 7.5 μm to 15 μm in size at 0.5 – 1.0 m. This is related to the property of kaolin which can flocculate, which is a process in which a solid form suspension in the form of flakes (pieces with larger dimensions).

The results of measuring the total pore diameter of the soil from the SEM photomicrograph on the BH02 soil profile of the Cijengkol slope are shown in the following figure. The pore diameter and the number decreases with the depth of the soil layer. The results of measuring the total pore diameter of the soil from the SEM photomicrograph on the BH02 soil profile of the Cijengkol slope are shown in the following figure. The pore diameter and the number decrease with the depth of the soil layer.

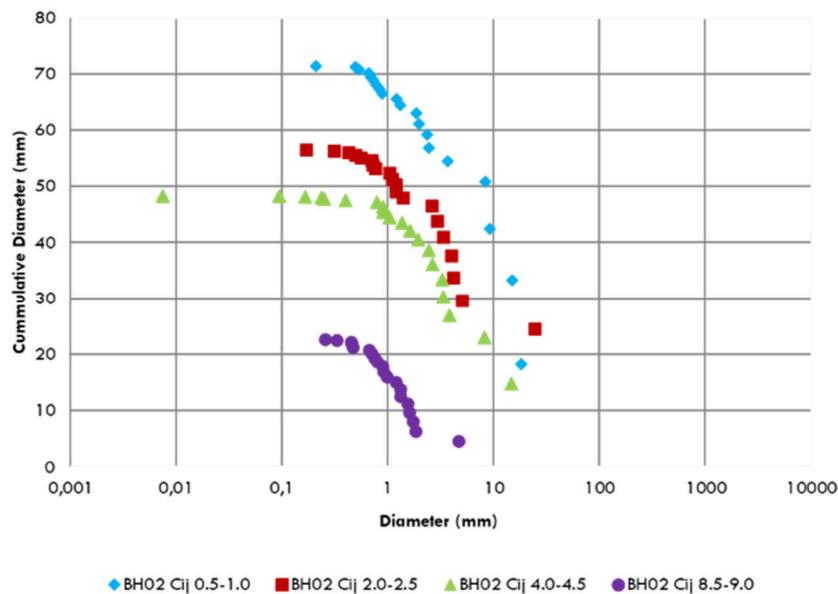


Fig. 16. Pore Diameter per soil layer BH02 Neglajaya

5. Conclusions

Research on the composition of residual trophic soil minerals carried out by XRD and SEM testing resulted in the following conclusions:

- In general, the same dominant type and mineral content are found on the slopes of volcanic tropical residual soils, namely Halloysite and Kaolinite
- Typical mineral profiles encountered along the soil profile are as follows:
 - a. On the surface of the soil there are minerals Goethite and Hematite
 - b. In deeper soil layers, alkaline minerals are found, namely carbonate and chlorite
 - c. In the next layer of soil, Feldspar minerals are found in large quantities.
- Because there are unique minerals at each depth of soil weathering, zone classification can be done easily starting from the Initial Leaching Zone at the deepest part where there is Feldspar, the Mineral Leaching Zone there is Carbonate and Chlorite and the Oxidation Zone on the surface (there are Oxide and Hydroxide minerals). So that it can form a Residual Tropical Volcanic Weathering Profile
- The SEM study showed that early signs of differences in the physical properties of the residual soil, namely differences in the shape and size of the soil chips and differences in the size of the soil pores.

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