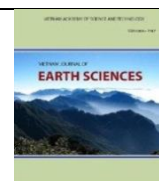




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## “Garnierite” in weathering crust of ultramafic blocks from Cao Bang area, north Viet Nam

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### ABSTRACT

“Garnierite” is an important group of ore minerals in the supergene nickel deposits. Garnierite group minerals are Ni-bearing hydrous magnesium silicate formed due to the lateritic weathering of ultramafic rocks under humid tropical conditions. In the weathering crust on ultramafic blocks Ha Tri and Phan Thanh (Cao Bang province), garnierite group minerals have been found in the saprolite zone or as a coating on the surface of the fissures. Analytical results show that garnierite consists of a mixture of three solid solutions: (i) serpentine-like - 7 Å (lizardite-nepouite), (ii) talc-like - 10 Å (pimelite-willemsite) and (iii) chlorite-like - 14 Å (clinochlore-minite). EPMA analysis shows that garnierite in Cao Bang area is characterized by high Ni content (25.50–40.06%), low Fe content (0.09–0.9%) and almost no Al (<0.02%), similar to garnierite in the famous nickel mines in New Caledonia, Dominican Republic, etc... Notable, Ni and Mg contents in garnierite show a clear negative correlation, indicating the replacement for each other in the phases. Under the scanning electron microscope (SEM), garnierite has a raspberry shape forming continuous zones covering the unfinished weathered rock surface. These zones represent different stages of garnierite formation. The results of the study allow proposing a model of garnierite formation in the lateritic weathering zone in ultramafic blocks in the Cao Bang area.

*Keywords:* Garnierite; nickel laterite; hydrous magnesium silicate deposits; weathering of ultramafic rocks; Cao Bang area; north Vietnam.

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### 1. Introduction

Nickel laterite is loose materials formed during weathering of ultramafic rocks under humid tropical conditions (Trescases, 1975; Golightly, 1981; Brand et al., 1998). It is an important source of Ni, accounting for 60% of the world's nickel resources (Kuck, 2013). Based on the composition of the major Ni-

bearing minerals, nickel laterite ore is usually divided into 3 types: (i) Oxide (oxide laterite deposits), of which the ore minerals are mainly oxyhydroxides Fe; (ii) Clay silicate (clay silicate deposits) that is dominated by Ni-rich smectites; and (iii) Hydrous magnesium silicate (hydrous magnesium silicate deposits) in which the ore is mainly Mg-Ni phyllosilicates (including garnierites) (Villanova-de-Benavent, 2014).

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The term “garnierite” is used to refer to the special lateritic Ni ore of grass-green color, poor crystallinity, and fine-grained nature, and it often occurs in lateritic Ni deposits of the hydrous Mg silicate type (Brand et al., 1998; Golightly, 2010; Butt and Cluzel, 2013). Due to its high Ni content, garnierite is an important nickel ore in many famous supergene deposits in the world such as the Goro deposit, New Caledonia (eg, Wells et al., 2009), the Sorowako deposit, Indonesia (eg, Sufriadin et al., 2015; Ilyasa et al., 2016), the Falcondo deposit, Dominican Republic (eg, Tauler et al., 2009; Villanova-de-Benavent et al., 2014). Some miners call them “green gold” and consider them a reliable indicator to find Ni ore in laterite because of its outstanding color and distinctive appearance (Wei Fua et al., 2018).

Garnierite group minerals have been identified as a series of layer Mg silicates including serpentine, talc, sepiolite, chlorite and smectite (Manceau and Calas, 1985; Brindley and Hang, 1973; Brindley et al., 1978; Faust, 1966; Manceau and Calas, 1985; Springer, 1974; Soler et al., 2008; Talovina et al., 2008; Wells et al., 2009; Wei Fua et al., 2018). Although garnierite is not recognized by the International Mineral Society (IMA) as a mineral, this is a convenient term used by mining geologists to give all green Ni silicates when it is not possible to more fully characterize (Brindley, 1978).

In Vietnam, there were some preliminary descriptions of garnierite in weathering crusts of Nui Nua (Thanh Hoa) and Ban Phuc (Son La) ultramafic blocks (Nguyen Khac Giang, Pham Van An, 1998). However, the determination of mineral phases of the garnierite group was based only on the Pelletier classification diagram and there is no data of another crucial analysis. The occurrence of garnierite in ultramafic blocks in Cao Bang area was not recognized in the above-mentioned study.

Recently, Nguyen Van Pho and co-authors (Nguyen Van Pho et al., 2018) have discovered the occurrence of supergene nickel minerals, including garnierite in weathering profiles of ultramafic massive Ha Tri (Cao Bang). However, in this work, the authors do not have detailed studies on mineral phases as well as their chemical composition. In this paper, we present the latest study results including a detailed analysis of mineral phases as well as the chemical composition of garnierite in Ha Tri and Phan Thanh ultramafic blocks (Cao Bang province) and provide a model of garnierite formation in this area.

## 2. Geological setting

The Ha Tri and Phan Thanh ultramafic intrusive blocks are considered as part of the Permian-Triassic Cao Bang complex (Halpin et al., 2016; Tran et al., 2008) occurred in Song Hien tectonic structure, Northeast Vietnam (Fig. 1). Phan Thanh block is about 2.5 km long and 0.1–0.3 km wide, located southeast of the Suoi Cun intrusive block - the biggest one of the Cao Bang complex and consists of melano-gabbro and plagioclase-bearing olivine lherzolite (Svetlitskaya et al., 2017). The Ha Tri block is a small body with a length of about 4.6 km and a width of 0.3–1.0 km, located in the southeast of the Phan Thanh block and consists of sulfide-bearing olivine melano-gabbro and plagioclase-bearing lherzolite (Svetlitskaya et al., 2017). Mineral compositions of Ha Tri and Phan Thanh blocks are similar and include olivine ranged from 40 to 70% or more, clinopyroxene about 15 to 20%, orthopyroxene is of a few percents, plagioclase is up to 10%. Minor minerals include Cr-spinel and reddish-brown phlogopite. The ore minerals are magnetite, ilmenite, and sulfides (pyrrhotite, violarite, chalcopyrite and pyrite). Secondary minerals include hornblende, chlorite and serpentine. Olivine is often serpentized strongly or completely (Nguyen Van Pho et al., 2018).

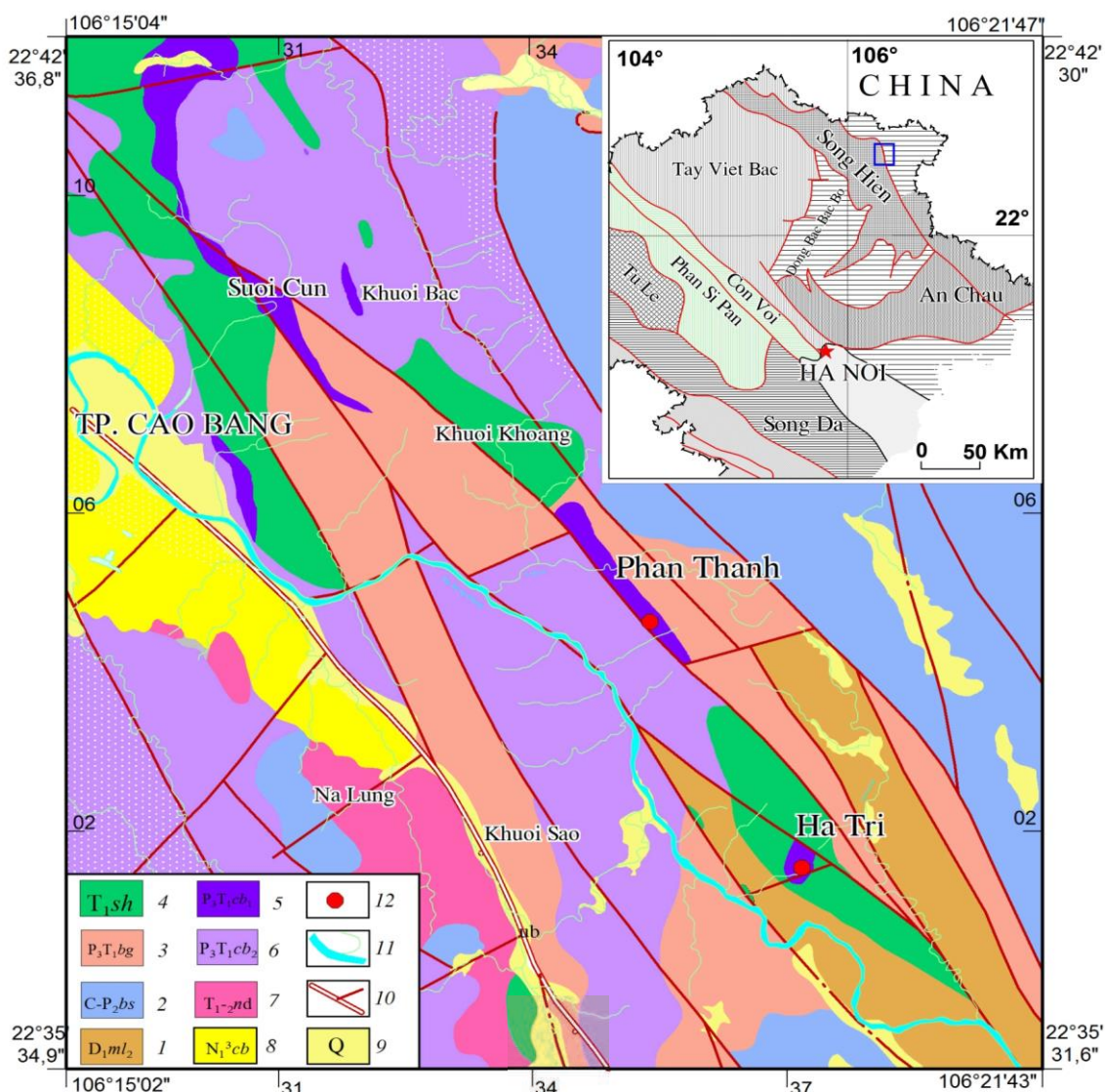


Figure 1. Geological map of Ha Tri and Phan Thanh ultramafic blocks, Cao Bang province. 1. Mia Le Formation ( $D_1ml$ ); 2. Bac Son Formation ( $C-P_2bs$ ); 3. Bang Giang Formation ( $P_3-T_1bg$ ); 4. Song Hien Formation; 5-6. Mafic-ultramafic magmas of Cao Bang Complex: 5. Phase 1 ( $vP_3T_1cb_1$ ), 6. Phase 2 ( $\sigma P_3T_1cb_2$ ); 7. Granite of Nui Dieng Complex ( $\gamma nT_1-2nd$ ); 8. Cao Bang Formation; 9. Quaternary sediments; 10. Faults; 11. Rivers and streams; 12. Location of the study area. (modified after Geological maps of scale 1:50.000, Trung Khanh sheet (Nguyen Cong Thuan et al., 2005) and Cao Bang - Dong Khe sheet (Nguyen The Cuong et al., 2000))

The thickness of weathering crust in Ha Tri and Phan Thanh blocks varies depending on terrain and generally ranges from 5 to 10m in high mountainous areas, while in lower areas, the thickness may exceed 20m. The

weathering profile can be divided into two main zones: the upper limonite zone and the lower saprolite zone, distinguished by changes in the color and structure of the rocks (Fig. 2a). These zones may not exist fully in



all weathering profiles due to erosion or specific weathering conditions.

In the weathering profiles, the garnierite was found only in joints and fractures cut through unfinished weathered ultramafic rocks in the middle of the saprolite zone (Fig. 2b, c), usually in the form of veins that

fill the fissures. The thickness of the garnierite veins varies from a few millimeters to centimeters and the length can range from a few centimeters to tens of centimeters. Besides veins, garnierite also appears as thin coatings on the surface of fissures with a thickness of several millimeters (Fig. 2c).

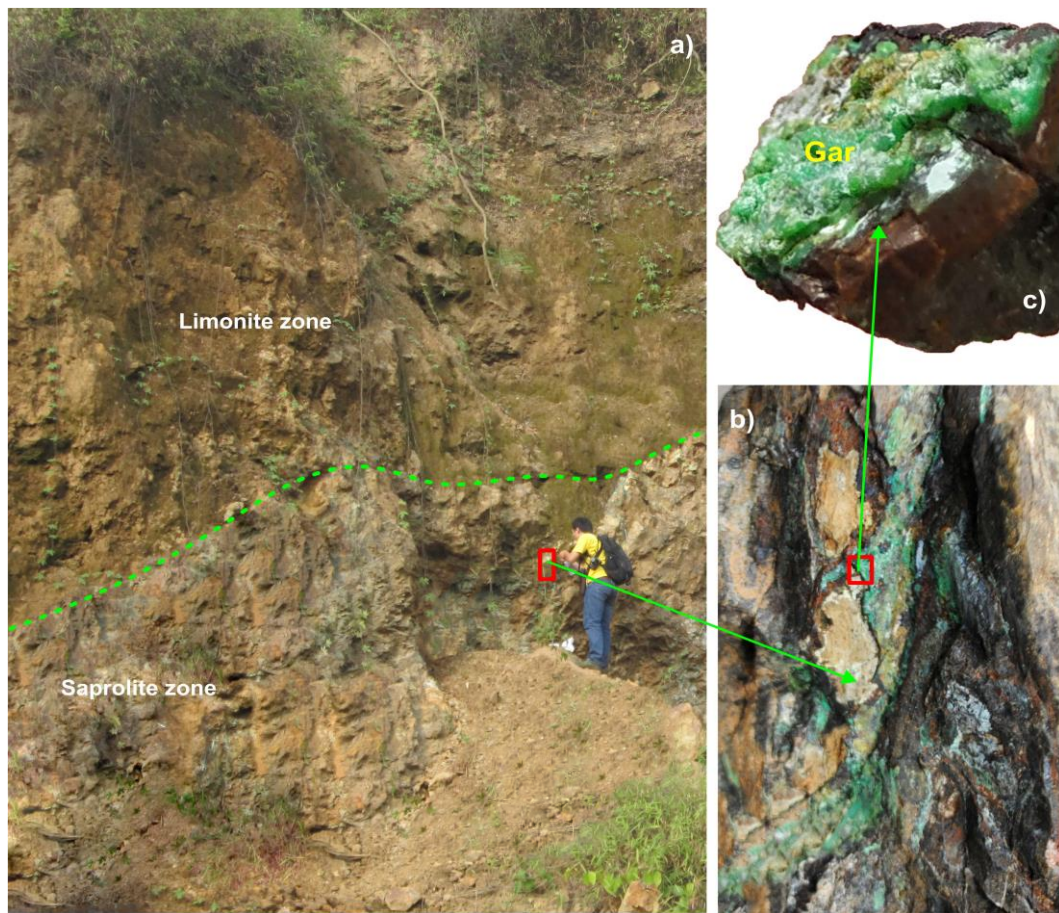


Figure 2. (a) Weathering profile in ultramafic rocks of Ha Tri block; (b, c) Garnierite appears as thin coatings on the surface of fissures (Gar-garnierite)

### 3. Samples and Analytical methods

A total of seven samples were collected from the saprolite zone in the study area, including four garnierite samples (G-1, G-2, G-3, G-4) in Ha Tri block and three samples (G4, G5, G6) in Phan Thanh block. All samples were analyzed using multiple methods, including X-ray powder diffraction

(XRD), scanning electron microscopy (SEM-EDX), and electron Probe Micro-Analysis (EPMA). The whole analyses were carried out in the Institute of Geological Sciences, VAST.

The mineralogical composition of powdered samples with  $<40 \mu\text{m}$  size fractions of the garnierite was investigated using a PANalytical Empyrean diffractometer,



working on Cu target at 45 kV and 40mA. The range of  $2\theta$  scanning was from  $5^\circ$  to  $65^\circ$ .

Garnierite mineral morphology was examined by Scanning electron microscopy (SEM), and the elements content was determined using an energy dispersive spectrometer. Instruments used were a Quanta 650 – FEI, operating at 5–30 kV accelerating voltage,  $3 \times 10^{-12}$  A primary beam current, and 5–15 mm working distance.

The EMPA analysis was performed on polished sections to determine the chemical composition of minerals using SXFive electron microprobe with a wavelength dispersive system. The measurement conditions were 20 kV accelerating voltage, 15 nA probe current, and 2  $\mu\text{m}$  beam diameter.

#### 4. Results

The X-ray powder diffractograms of G1

and G2 samples have the maximum diffraction peak at  $14.34 \text{ \AA}$ , which coincides with the characteristics of the chlorite mineral group, including the minerals in the clinocllore-minite isomorphic series  $[(\text{Ni},\text{Mg},\text{Al})_6(\text{Si},\text{Al})_4\text{O}_{10}(\text{OH})_8]$ . The diffractograms of samples G5, G6, G7 have the maximum diffraction peak at  $9.9 \text{ \AA}$ , corresponding to the talc mineral group, including the minerals in the talc - willemseite  $[(\text{Ni},\text{Mg})_3\text{SiO}_4\text{O}_{10}(\text{OH})_2]$  isomorphic series. Samples G3 and G4 have the maximum diffraction peak at  $7.29 \text{ \AA}$ , corresponding to the characteristics of the minerals of the serpentine group, which includes the minerals in the lizardite-nepouite  $[(\text{Ni},\text{Mg})_3\text{Si}_2\text{O}_5(\text{OH})_4]$  isomorphic series. The X-ray diffraction spectrum of garnierite samples from Cao Bang area is summarized in Fig. 3.

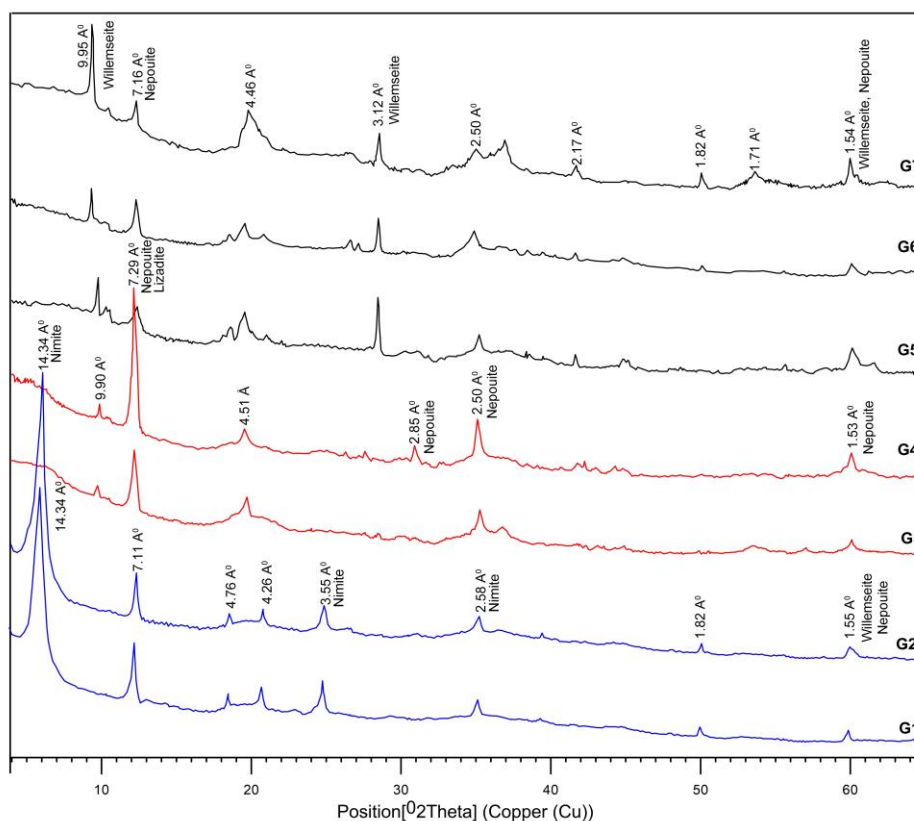


Figure 3. X-ray powder diffractograms of Garnierite from Cao Bang

The chemical composition of garnierite is summarized in Table 1. Garnierite has the  $\text{Fe}_2\text{O}_3$  (total Fe) from 0.02 to 0.91% and  $\text{NiO}_2$  concentration ranged from 25.50 to 40.06%,  $\text{MgO}$  from 6.41 to 19.57%, and  $\text{Al} < 0.02\%$ .

Table 1. Major element composition \*(wt.%) of garnierite from Cao Bang area

Sample	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	$\text{Cr}_2\text{O}_3$	$\text{NiO}$	$\text{MgO}$	$\text{CaO}$	Total
G1	22.28	0.02	0.23	0.00	37.13	14.85	0.02	74.53
G2	24.09	0.02	0.27	0.00	31.61	19.57	0.02	75.58
G3	25.42	0.02	0.83	0.01	33.64	15.70	0.03	75.65
G4	30.00	0.01	0.91	0.01	25.50	19.50	0.02	75.94
G5	33.65	0.02	0.1	0.00	40.06	6.41	0.06	80.30
G6	34.11	0.02	0.09	0.00	37.28	7.93	0.05	79.48
G7	35.41	0.01	0.12	0.00	37.05	9.88	0.06	82.53
Gar1-a2	37.89	0.01	0.02	0.01	46.06	5.25	0.01	89.25
P-Green	38.49	0.03	0.12	0.00	49.65	2.27	0.01	90.57

Note : \*Analyzed by EPMA; Samples G1-G7 from Cao Bang area (this study); Gar1-a2: Dominican Republic (Cristina Villanova-de-Benavent., 2014); P-Green: New Caledonia (Michel Cathelineau et al., 2015)

The results of SEM analysis with magnifications of 800x and 1000x; show that the garnierite of the Cao Bang area appears to

have a raspberry form, forming continuous zones that covered on surface of unfinished weathered ultramafic rocks (Fig. 4).

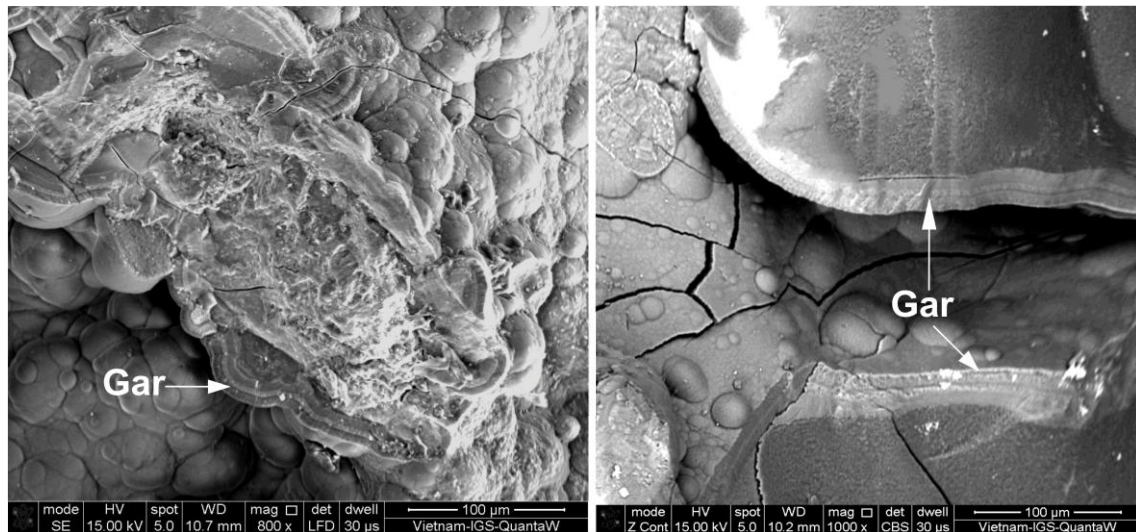


Figure 4. SEM-photomicrographs of garnierite from Cao Bang (Gar-garnierite)

## 5. Discussions

X-ray diffraction analysis shows that the garnierite from Cao Bang area has the main diffraction peaks at  $\sim 14 \text{ \AA}$ ,  $\sim 10 \text{ \AA}$  and  $\sim 7 \text{ \AA}$  which are characteristic for the structure of chlorite, talc and serpentine minerals (Fig. 3). However, in Talc garnierite samples (G5, G6,

G7), apart from the typical peaks at  $\sim 10 \text{ \AA}$ , diffraction picks  $\sim 7 \text{ \AA}$  also appear to be typical for serpentine-type (with weaker intensity) and vice versa. In serpentine-type garnierite samples (G3, G4), there also appeared diffraction peaks  $\sim 10 \text{ \AA}$  with weaker intensity, which is typical of talc-type garnierite (with weaker intensity). This

indicates that the garnierite group minerals in the Cao Bang area exist as a mixture of talc, serpentine and chlorite-type phases (Villanova-de-Benavent et al., 2014).

As shown in the Si-Mg-(Ni+Fe) classification diagram by Brand et al., 1998, the samples G5, G6 and G7 fall into willemseite field and belong to the talc-type garnierite series (Fig 5). Both G3 and G4 samples belong to the serpentine-type garnierite series, but G3 falls into Ni-lizadite field and G4 into nepoulite field. Sample G1 distributes in nimite field while G2 falls into nickeloan chlorite field (see Fig. 5 again). Distribution of samples in Si-Mg-(Ni+Fe) diagram is completely accordant with the results of XRD mineral phase analysis, that the mineral of garnierite group from Cao Bang consists of serpentine-, talc- and chlorite- types.

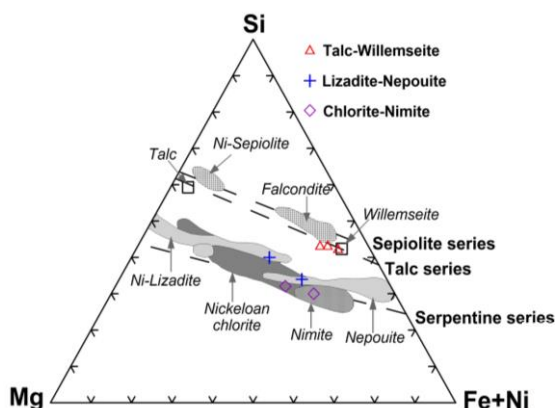


Figure 5. Si-Mg-(Ni+Fe) ratios for garnierite in Cao Bang. (Compositional fields are taken from Brand et al., 1998)

The composition of garnierite from Cao Bang area is characterized by high Ni content (25.50–40.06%), low Fe content (0.09–0.9%) and almost no Al (<0.02%), similar with garnierite from famous nickel deposits in the world such as New Caledonia, Dominican Republic and many others. Notably, Ni and Mg contents have a clear negative correlation, indicating the replacement for each other in the mineral phases (Fig. 6). The negative

correlation of Ni and Mg is even more evident when using the line scan mode in SEM-EDX to observe the variation of Ni and Mg content along the chosen cross-section (A-B) from garnierite zone cut through unfinished weathered rock (Fig. 7). In the outer garnierite zone, the Ni content increases while the Mg content decreases, in the opposite, when cutting through unfinished weathered rocks, the Ni content decreases correspondingly to the increase of the Mg content. This suggests that Ni replaced Mg in the mineral structure and maximum enrichment of Ni happened when Mg was completely replaced. The mentioned correlation of Ni and Mg content completely coincides with the previous results of Nguyen Van Pho et al. (2018) and confirmed by EPMA analysis presented above (Fig. 6, Table 1).

Under the scanning electron microscope (SEM), the garnierite in Cao Bang area forms consecutive zones on the unfinished weathered ultramafic rock (Fig. 4), which shows that garnierite is formed by several different stages (Villanova-de-Benavent et al., 2014; Wei Fua et al., 2018).

From the results presented above it is possible to propose a garnierite formation model in the lateritic weathering zone of ultramafic rocks in Cao Bang area as follows (apply Wei Fu's model, 2018):

As the most of garnierite ores in other deposits in the world, garnierite in Cao Bang area is formed and accumulated due to humid tropical weathering of ultramafic rocks under favorable conditions (tectonic rifting, low groundwater levels, etc...) (Butt and Cluzel, 2013; Brand et al., 1998; Elias, 2002; Golightly, 1981). During the lateritization process, the main Ni-bearing silicates are altered and transformed into more stable secondary phases (minerals) under oxidative conditions. Under these conditions, olivine is considered the least stable mineral and the first mineral to be weathered by hydrolysis according to the following reaction (reaction 1) (Freyssinet et al., 2005):



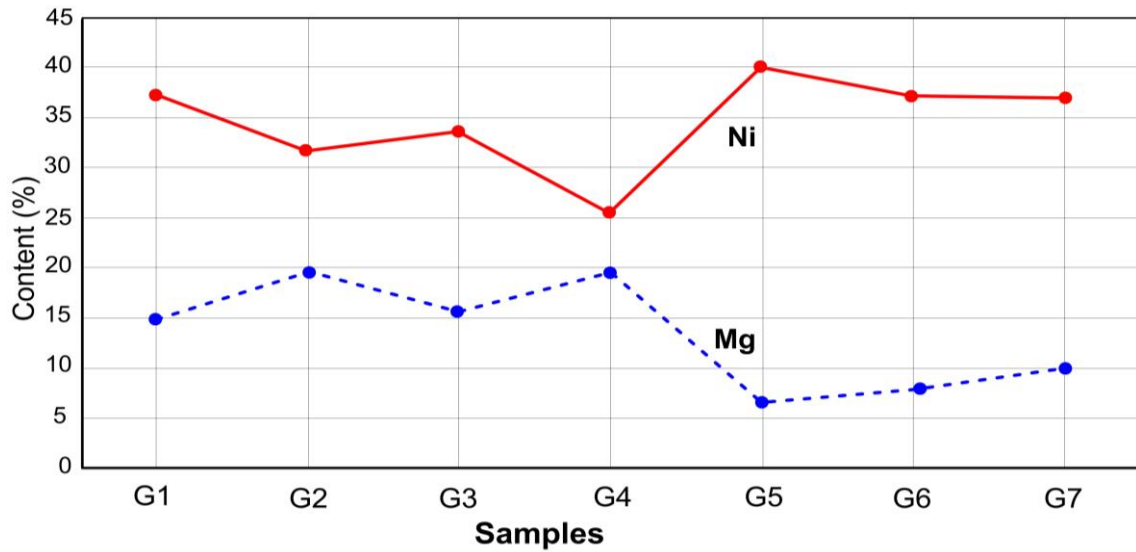


Figure 6. Ni and Mg contents of Garnierite from Cao Bang (EPMA data)

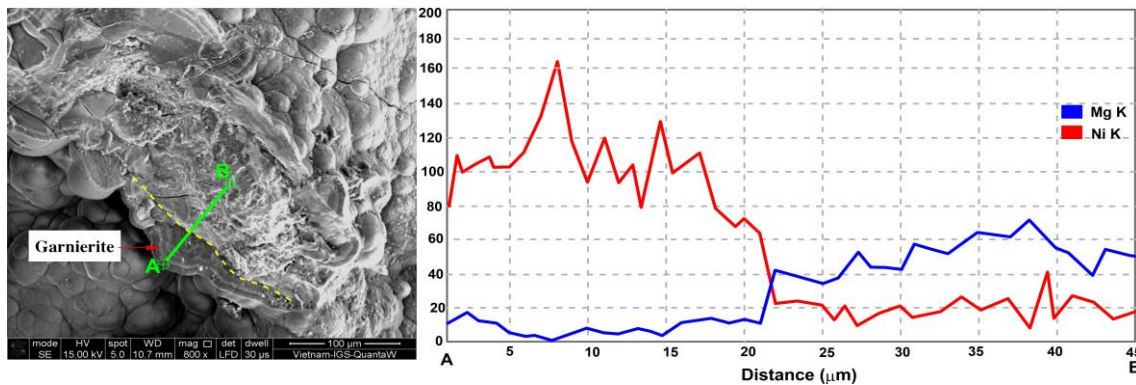


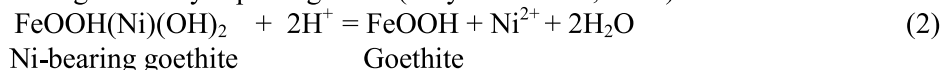
Figure 7. Variation of Ni and Mg contents along cross-section A - B cutting through the boundary between garnierite zone and unfinished weathered rock (using SEM-EDX detector)



The Mg is then dissolved and transported by weathered solution, while  $\text{Fe}^{2+}$  is oxidized to insoluble  $\text{Fe}^{3+}$  and precipitated as goethite in the limonite zone (Golightly, 2010). Ni released from Ni-bearing silicates (mainly from olivine) is retained in goethite by replacing Fe

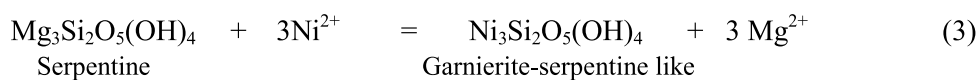
or being adsorbed. Fe retention in limonite zone explains the very low Fe content in garnierite minerals in Cao Bang area.

As weathering continues, the adsorbed Ni on goethite can be released by reaction 2 (Freyssinet et al., 2005):



The liberated Ni from goethite is first incorporated in Ni-containing serpentine (Pelletier, 1996) to form serpentine-type

garnierite with the mechanism to replace Mg according to reaction 3 (Freyssinet et al., 2005):



After Ni-serpentine reaches a saturation state, excess Ni is precipitated in the form of Ni-Mg silicates (garnierite) in open spaces (fissures, fractures) near the water table (Freyssinet et al., 2005). This process depends on the pH of the weathering solution. When the pH dramatically changes from acidic to alkaline environment garnierite will begin to precipitate (Golightly, 1979).

So how can the elements form garnierite (Ni, Mg, Si,...) move through all the weathering profile to the bottom of the saprolite zone and precipitate into garnierites? According to Wei Fua et al, 2018, the decisive factor is the "favorable flow" in the weathering section, it can be the

big pore system created by the combination of biological factors (tree roots, wormholes, etc.) and geological factors (faults, fissures, fracture zones, etc.). Under humid rainy tropical conditions, "favorable flows" act as a conduit for the transport of Ni as well as weathering solutions to deeper zones and lead to precipitation of garnierite there. According to this view, garnierites cannot be present in the entire weathering zone, they only form in some places where favorable flows occur (fissures, faults, etc.) (Fig. 8). This is entirely suitable for detecting garnierite only in the form of fissure filling or thin coatings on fissures and faults in Cao Bang area.

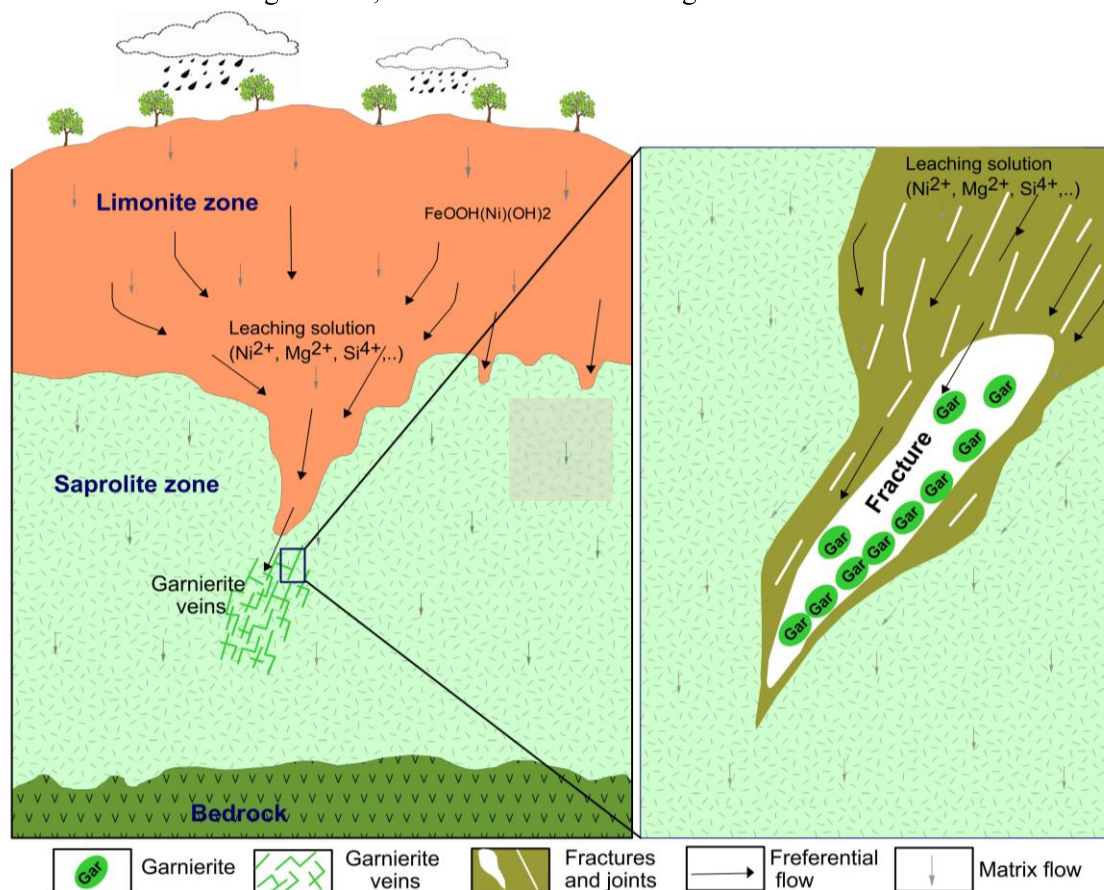


Figure 8. Model of garnierite formation in Cao Bang area (after Wei Fu et al., 2018), Gar-Garnierite

## 6. Conclusions

Cao Bang garnierite consists of a mixture of three types: (i) serpentine type (lizardite-nepouite) - 7 Å, (ii) talc type (pimelite-willemseite) - 10 Å and (iii) chlorite type - (clinochlore-minite) - 14 Å.

Garnierite in the Cao bang area is characterized by high Ni (25.50–40.06%), low Fe (0.09–0.9%) content and almost no Al (<0.02%). The Ni and Mg have a clear negative correlation, indicating the exchange with each other in forming phases (minerals). The garnierite is formed in different stages.

The garnierite formation in the weathered section of Cao Bang area starts from the release of Ni from the parent rock (mainly from olivine) during the lateritic weathering process. Ni then moves to the lower part of the weathering profile through the favorable flow system (fracture, fissures, tectonic faults, etc.) and form serpentine-type garnierite by replacing Mg in the first stage. As Ni-serpentine reaches a saturation state, excess Ni is precipitated in the form of layer Ni-Mg-silicates (Talc-and chlorite-type) in open space (fractures, fissures, fault).

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