Deposit potential and distribution of cobalt and nickel in the sludge of chromite placer mining process at Nui Nua ultramafic massif area, Thanh Hoa province, Vietnam

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Abstract: Chromite placer is widely distributed in Quaternary sediments located around the Nui Nua ultramafic massif in Thanh Hoa province, Vietnam. The long-term mining works targeting chromite placer in this area have left a huge amount of waste sludge, up to tens of millions of tons. The paper aims to introduce a deposit potential and distribution of cobalt- and nickel- bearing minerals within the waste sludges generated from the chromite placer mining operations around the mines situated near the Nui Nua ultramafic massif in Thanh Hoa province, Vietnam. Based on field investigations and sample analyses, two groups of waste sludge have been identified: clay-sized sludges and debris waste sludges. The former group is plastic bentonite clay, distributed in depressions and low terrain, and has low contents of cobalt (0.06%) and nickel (0.67%), which is of impractical significance for the recovery purposes of cobalt and nickel. The latter group is usually distributed in relatively high terrain, forming waste sludge ranging from several thousand to hundreds of thousands of tons in volume. These deposits include rock fragments, mineral fragments derived from the Nui Nua ultramafic massif, along with dark spherical nodules rich in goethite, limonite, and Fe-Mn hydroxides, which contain relatively high contents of cobalt (up to 0.75%) and nickel (up to 2.43%) in the waste sludge and chromite placer mines. The cobalt and nickel are mainly concentrated in Fe-Mn nodules made up of goethite, limonite, todorokite, and other Fe-Mn hydroxides. Data from chromite placer explorations, combined with the study results, provide reliable insights into the distribution of cobalt and nickel resources within the waste sludges and chromite placer deposits in the Co Dinh area (northeast of Nui Nua ultramafic massif) and Mau Lam (southwest of Nui Nua massif), Thanh Hoa province. A preliminary estimation of potential resources of Co and Ni metals have been made for the debris waste sludges at Co Dinh and Mau Lam areas based on the ratios of debris particles in the sludges and their contents of Co and Ni along with published resources and reserves of chromite placer ores.

Keywords: nickel, cobalt, waste sludge, chromite, Nui Nua ultramafic, Thanh Hoa – Vietnam

INTRODUCTION

Nickel (Ni) and cobalt (Co) metals play an increasingly important role in modern life, especially in the field of electric vehicle production. The demand for the use of Ni and Co in the production of batteries and motors for electric vehicles has skyrocketed in recent years and is expected to increase greatly in the future (Shedd 2015, 2018, Tisserant & Pauliuk 2016, Hitzman et al. 2017, Wilson 2017, Leighton 2021). Previously, Ni and Co ores were predominantly mined from sulfide mines, but in recent decades there has been a significant shift towards laterite (oxides and silicates) deposits (Butt & Cluzel 2013, Marsh et al. 2013, Mudd & Jowitt 2013, Putzolu et al. 2020).

History of geological and mining works at Nui Nua ultramafic massif

The Nui Nua ultramafic massif in Thanh Hoa province and its associated chromite placer mines containing Co and Ni metals as by-products have been extensively described in geological literature (e.g. Tan 1956, Vietnam Geological Department 1963, Chien 1964, Quan 1980, Lam 1983, Dao 1983, 1996, Giảng & An 1998, Tuan 2012, Ha et al. 2019). Quaternary sediments with thicknesses varying from 1–2 m to several tens of meters are distributed around the base of the Nui Nua ultramafic massif. Chromite placer ore bodies, along with occurrences of Ni and Co, have been identified within the Quaternary sediments (Tan 1956, Vietnam Geological Department 1963, Lam 1983, Dao 1983, 1996).

To the northeast of the Nui Nua ultramafic massif, there are chromite placer mining sites such as Co Dinh, My Cai, Hoa Yen, Tinh Me, and An Thuong, collectively referred to hereafter as Co Dinh area, covering a total area of up to 65 km^2. Similarly, to the southwest of the Nui Nua massif, chromite mine sites like Mau Lam and Bai Ang are collectively referred to as the Mau Lam area, with an area of nearly 6 km^2.

Chromite placer mines at the Nui Nua ultramafic massif were discovered by French geologists in 1927. Between 1954 and 2011, numerous surveys and explorations of chromite ore deposits in the area were conducted, with contributions of worker from China and the former Soviet Union (Tan 1956, 1962, Vietnam Geological Department 1963, Lam 1983, Dao 1983). During a period from 1930 to 1931, the French extracted 4,231 tons of crude ore, whilst from 1942 to 1944, the Japanese recovered 12,377 tons of chromite ore. In the Co Dinh area, the Co Dinh Chromite Joint Stock Company has been mining chromite ore in an area of 16.6 km^2 since 1996. In the Mau Lam area, the Thanh Hoa Mining, Processing and Exporting Mineral Joint Stock Company has been extracting chromite over an area of 1.6 km^2 since 1997.

Data on resources and reserves of Co and Ni have been reported, but with low concentrations of Ni (averaging 0.5–0.8%) and Co (0.018–0.054%) (Vietnam Geological Department 1963, Lam 1983, Dao 1983).

Geological setting of Nui Nua ultramafic massif and surrounding chromite mining sites

Nui Nua ultramafic massif

The geology of the Nui Nua ultramafic massif and adjacent areas shown in Figure 1. The massif extends northwest-southeast for about 15 km, with a width ranging 1–7 km (making it is the largest ultramafic massif in Vietnam). Rocks surrounding Nui Nua massif include Paleozoic quartz-sericite schists, green schists, and Mesozoic aged coarse-grained sediments, and rhyolite. The Co Dinh and Mau Lam areas are covered by Quaternary sediments with thicknesses ranging from 5–7 m to 60–70 m. The Nui Nua massif is intersected by many fault systems and is cut through by diabase and gabbro-diabase veins which are 470 million years old (Hieu et al. 2014).

The rocks of the Nui Nua ultramafic massif consist mainly of harzburgite, dunite, lherzolite, and serpentinite. Harzburgite is primarily distributed in the central and southeastern parts of the massif (Mau Lam area), while serpentinite is widely distributed in both the Co Dinh area (My Cai site) and Mau Lam area (Bai Anh site) (Fig. 1). According to Giảng and An (1998) and Dang (2020), the Nui Nua ultramafic rocks exhibit high MgO content (38.37–40.34%), relatively low Fe_2O_3 (6.43–7.24%), TiO_2 (0.01–0.02%), low Al_2O_3 (0.67–1.16%), and low $Na_2O + K_2O$ alkaline content (0.18–0.50%).

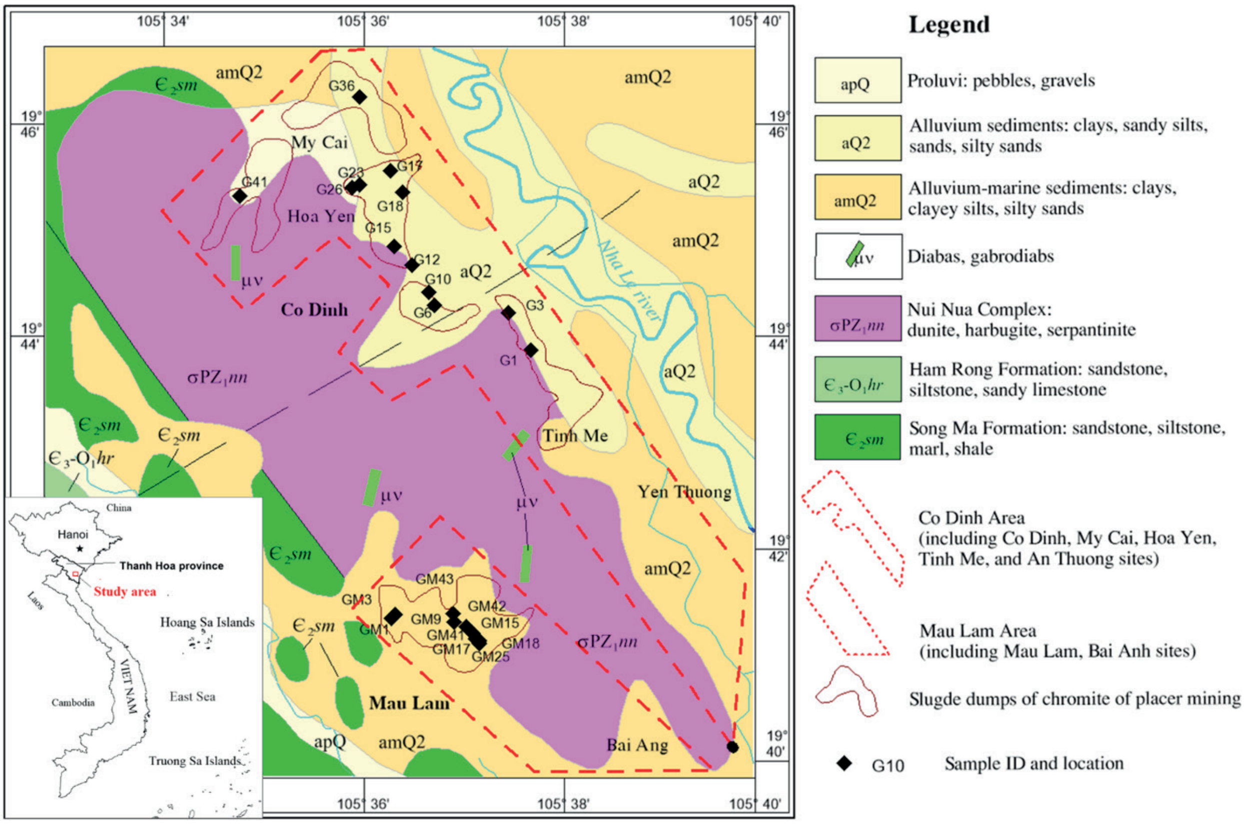


***Fig. 1.** Geological sketch map of Nui Nua ultramafic massif and locations of waste sludges at Co Dinh and Mau Lam area (modified form the Thanh Hoa geological map, scale 1 : 200,000)*

The weathering process at Nui Nua ultramafic massif shows zonation with relative enrichment of Ni and Co in the weathering crust compared with that in the host rocks. According to An and Giang (1997) and Giảng (2005) the weathering crust comprises two main zones: 1) saprolite zone, the largest volume at the slope of Nui Nua range, consists of smectite minerals (nontronite, saponite, ferrisaponite, montmorillonite), serpentine, hydrated serpentine, having Ni content of 0.3–0.7% and Co of 0.01–0.02%; 2) limonite zone, a smaller volume on the mountaintops of the Nui Nua range, includes limonite, goethite, and fewer clay minerals, having Ni content of 0.5–1.65% and Co of 0.01–0.07%. The type of weathering crust containing Ni and Co at Nui Nua massif exhibits many similarities to the weathering crust found on ultramafic massifs in Indonesia and worldwide (Berger et al. 2011, Farrokhpay et al. 2018).

Since 1930, the mining of placer chromite has led to the formation of large-scale waste sludges, distributed directly within the mining areas at the Co Dinh and Mau Lam areas (Fig. 1).

Quaternary sediments and Co, Ni-bearing chromite placer mines

In the Co Dinh area, a Quaternary sedimentary basin covering an area of 65 km^2 contains chromite ore bodies (Vietnam Geological Department 1963, Lam 1983). The Quaternary sediments hosting chromite placer are horizontally stratified, with thicknesses varying spatially of 15–70 m, locally up to 80 m. According to Lam (1983), the Quaternary sediments comprise two chromite ore layers: 1) lower chromite ore layer, at depth of 20–40 m (below ground level), consisting of pebbles, clay, chromite particles and Fe-Mn hydroxide nodules which are dark brown coloration (indicative of Mn richness) and brown colors (Fe richness), with Cr_2O_3 contents ranging 1.6–6.1% (averaged 3.4%), Fe-Mn nodules of 5–30% (averaged 15–20%); and 2) upper lens shaped chromite ore layer, at a depth of 2–3 m, with thicknesses of 1–10 m (averaged 4–7 m), consists of pebbles, clay, along with chromite particles and Fe-Mn nodules with Cr_2O_3 ranging 0.9–5.7% (averaged 3.2%). According to the Vietnam Geological Department (1963), the primary chemical component of the chromite ores in the Co Dinh area includes Cr_2O_3 of 4.36%, Ni of 0.48% and Co of 0.04%. Mn-Fe nodules are more abundant in the lower layer than the upper one. Besides clay minerals and Fe-Mn hydroxides, the ore layers also contain quartz, siderite, chromite, magnetite, ilmenite, and fragments of serpentinite.

In the Mau Lam area, the Quaternary sediments containing chromite and Fe-Mn nodules are distributed along the valley, with a length of 5–6 km and a width of 1–2 km (Fig. 1). The chromite-bearing sediments exhibit a relatively small thickness, ranging 2–10 m. According to Dao (1983, 1996) the sediments in the Mau Lam area have three layers, from bottom to top: 1) Layer 1 (0.4 m thick) including serpentinite fragments, a few quartz pieces, chromite, and Fe-Mn nodules; 2) Layer 2 (2.6 m thick) consisting of sandy clays, chromite particles, and Fe-Mn nodules; and 3) Layer 3 (2.1 m thick), comprising of dark grey clay, sand, pebbles. Layer 3 has a higher content of chromite ore and Fe-Mn nodules compared to Layer 1 and Layer 2. A geological comparison indicates that the three layers of sediments in the Mau Lam area correspond to Layer 1 at Co Dinh area.

The grain size distribution of chromite placer ores and waste sludge in the Co Dinh and Mau Lam areas is summarized in Table 1. The chromite ore bodies in Co Dinh and Mau Lam consistently contain Fe-Mn nodules, which are the important carriers of Ni and Co metals in the primary Ni and Co-bearing sediments as well as in chromite mining waste sludges.

The proportions of fragments with grain sizes larger than 0.074 mm are 49.0% and 64.9% in the Co Dinh and Mau Lam areas respectively (Table 1). The average proportions of grain size larger than 0.074 mm across the entire chromite mining in Co Dinh and Mau Lam is about 55%. This data is crucial for the estimation of potential sources of Ni and Co in the Co Dinh and Mau Lam areas.

The chromite mining activities surrounding the Nui Nua ultramafic massif has led to the formation of existing sludge dumps in the Co Dinh and Mau Lam areas. The materials of sludge dumps consist of either loose or adhesive, encompassing mud, clay, pebbles ranging from 0.01 mm to a few cm. Hereafter, they are collectively referred to as waste sludges. The waste sludges are widely distributed throughout the mining sites in the Co Dinh area (My Cai, Co Dinh and Tinh Me sites) and the Mau Lam area (Mau lam and Bai Ang sites) (Fig. 1). During the mining of chromite, Co and Ni were not of primary concern. However, they are selectively enriched in the waste sludges. forming potentially valuable secondary sedimentary ore bodies.

Table 1
Summary of particle composition of chromite ore bodies in the Co Dinh and Mau Lam (based on Tan 1962, Tuan 2012, Vietnam Geological Department 1963) areas

Grain size in diameter [mm]	Co Dinh area [%]	Mau Lam area [%]
>2.5	15.8	27.5
2.5–0.074	33.2	37.4
>0.074	49.0	64.9
<0.074	51.0	35.1

METHODS

Field investigation and sample analysis

The field investigation was meant to study the distribution and potential sources of Co and Ni in waste sludge and to collect samples of waste sludges. Depending on the distribution of materials in waste sludges, samples were collected on both the surface of the landfill and at depth with a weight of several kilograms. Samples were collected at waste sludges in the Co Dinh and Mau Lam areas (Fig. 1). In the laboratory, these samples were sieved and separated into particle size components for analysis: coarse particles (sizes greater than 0.074 mm in diameter) and fine particles (sizes smaller than 0.074 mm). The samples were further pulverized for XRD and ICP analyses. There were nineteen samples for grain size analyses.

Mineralogical and petrographic analysis

To study the mineral composition in primary ultramafic rocks, twenty samples were used for petrographic microscope analysis and other twenty samples were analyzed with a reflected light microscope.

The mineral composition of the coarse-grained samples was determined using stereo-microscope analysis (20 samples analysed). The mineralogical composition of fine grain samples was determined by means of the X-ray diffraction (XRD) method at the Geological Laboratory Analysis Center of the General Department of Geology and Minerals of Vietnam. Additionally, a scanning electron microscope (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS) equipment was used at the Centre for Advanced Technology Analysis, Hanoi University of Mining and Geology. XRD patterns of the samples were collected using a D8-Advance Bruker with Cu-Kα radiation (λ = 1.5406 nm) generated at 40 kV and 40 mA. The data were recorded in the Bragg angle (2θ) range of 3–70° with a scanning speed of 2° min^{-1}. Minerals were identified using the Evaluation 10.0 software with database (PDF-2 2004) provided by the International Centre for Diffraction Data. There were 25 samples analyzed with XRD. SEM and EDX (Quanta 450) were initially used to analyze the morphology of minerals and elements present in samples. The samples were polished and coated with carbon before analysis. A total of 36 samples were analyzed using SEM and EDX.

Chemical composition analysis

Samples were analyzed for the content of Co, Ni and associated metals (Fe and Mn) both in granulated particles and in iron-manganese nodules, utilizing atomic absorption spectroscopy (AAS) – PinAAcle 900T and inductively coupled plasma optical emission spectroscopy (ICP-OES) – Perkin Elmer Optima 8300. The analysis of samples was conducted at the Laboratory Analysis Center of the General Department of Geology and Minerals of Vietnam. The detection limits of the AAS – PinAAcle 900T systems equipment are 0.001% for Co, Ni, and Mn, and 0.01% for Total Fe. The samples were processed and analyzed in accordance with the Vietnamese National technical standards QCVN 53:2014/BTNMT for quality control on geological and mineral analysis results (MONRE 2014). A total of 150 samples from the Co Dinh and Mau Lam areas and an additional 5 samples taken at weathering crusts on the Nui Nua ultramafic massif are also used for ASS analyses.

RESULTS AND DISCUSSION

Formation, classification, and distribution of waste sludges

The waste sludges in the Co Dinh and Mau Lam areas were formed by the chromite placer mining activities in the areas (Figs. 1–3). Based on the grain size of particle compositions, the waste sludges are classified into two groups: clay-sized waste sludges and debris waste sludges.

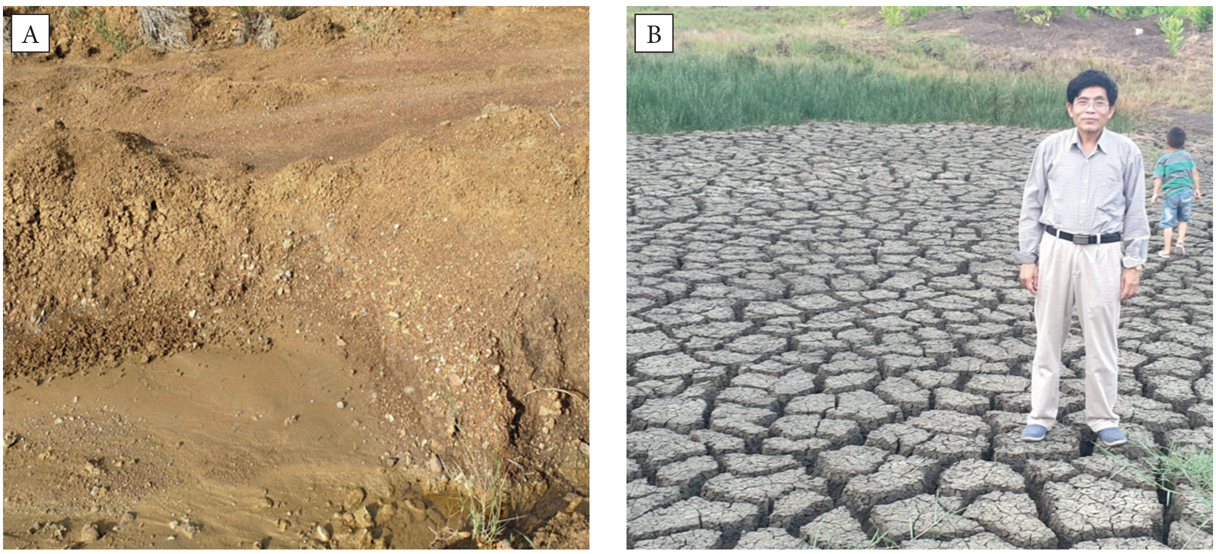


Fig. 2. *Clay sized waste sludges at Co Dinh area (A) and Mau Lam area (B)*

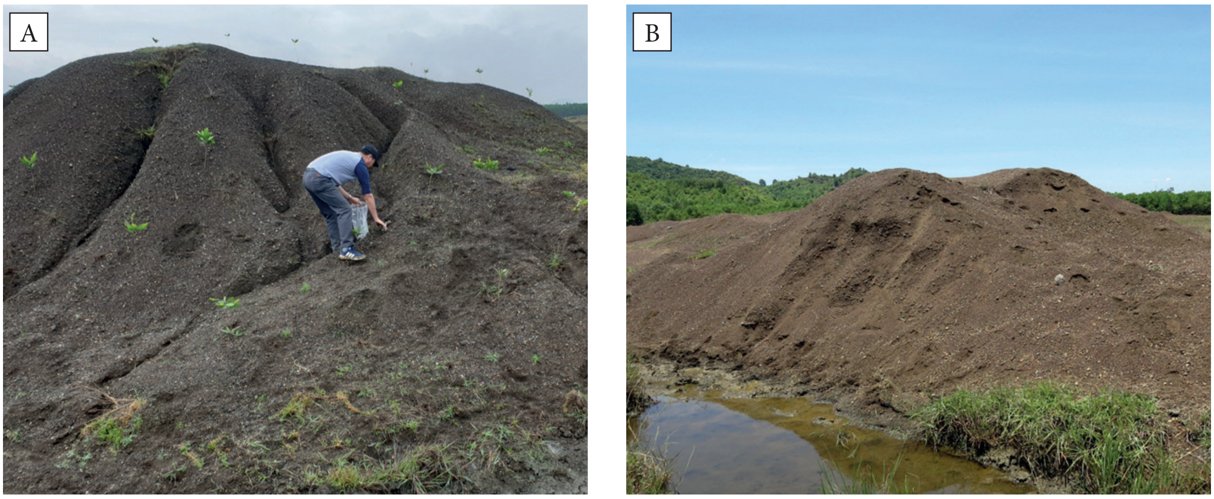


Fig. 3. *Debris waste sludges in the Co Dinh (A) area and Mau Lam area (B)*

The clay sized waste sludge predominantly consists of clay minerals fine grained particles. This group fills the low-lying terrain and excavated pits, forming accumulations of brown or bluish-grey bentonite clay, with a thickness from 1 to 4 m. Following dehydration, it becomes hard (Fig. 2).

The debris waste sludge: particle sizes ranging from 0.074 mm to 16 mm, accumulates into relatively large cone-shaped masses (Fig. 3). On-site, these masses of coarse-grained particle sludge are distributed around the mining ponds or on ground and forming chains of cone-shaped heaps. The height of these masses varies from 2–3 m to 9–10 m, covering an area of hundreds of square meters.

Mineral and chemical composition in the clay sized waste sludges

The petrographic and mineralogical characteristics

The XRD and SEM analyses indicate that the clay sized waste sludges primarily consist of the smectite group (mainly nontronite) (accounted for 32%), quartz (14%), goethite (14.5%), serpentinite group, including antigorite and lizardite (19%), and other minerals (less than 5%) of hematite, amphibolite, chlorite, etc. The clay sized waste sludges also include a trace of limonite, goethite, and todorokite (see Table 2, Figs. 4, 5 for example).

Table 2
Mineral compositions [%] of the clay sized sludge analyzed by XRD

Sample ID	Mineral composition [%]								
	todorokite	goethite	hematite	magnetite	quartz	chlorite	antigorite	amphibole	nontronite
Co Dinh area									
G10b	–	11–13	1–3	–	7–9	9–11	14–16	4–6	37–39
G17b	–	14–16	3–5	2–4	10–12	9–11	24–26	4–6	21–23
G26b	≤1	14–16	–	4–6	5–7	10–12	18–20	7–9	26–28
Mau Lam area									
GM1b	≤1	9–11	1–3	1–3	25–27	6–8	8–10	5–7	26–28
GM4b	≤1	13–15	1–3	–	16–18	5–7	6–8	1–3	44–46
GM18b	1–3	18–20	3–5	1–3	15–17	4–6	5–7	6–8	33–35

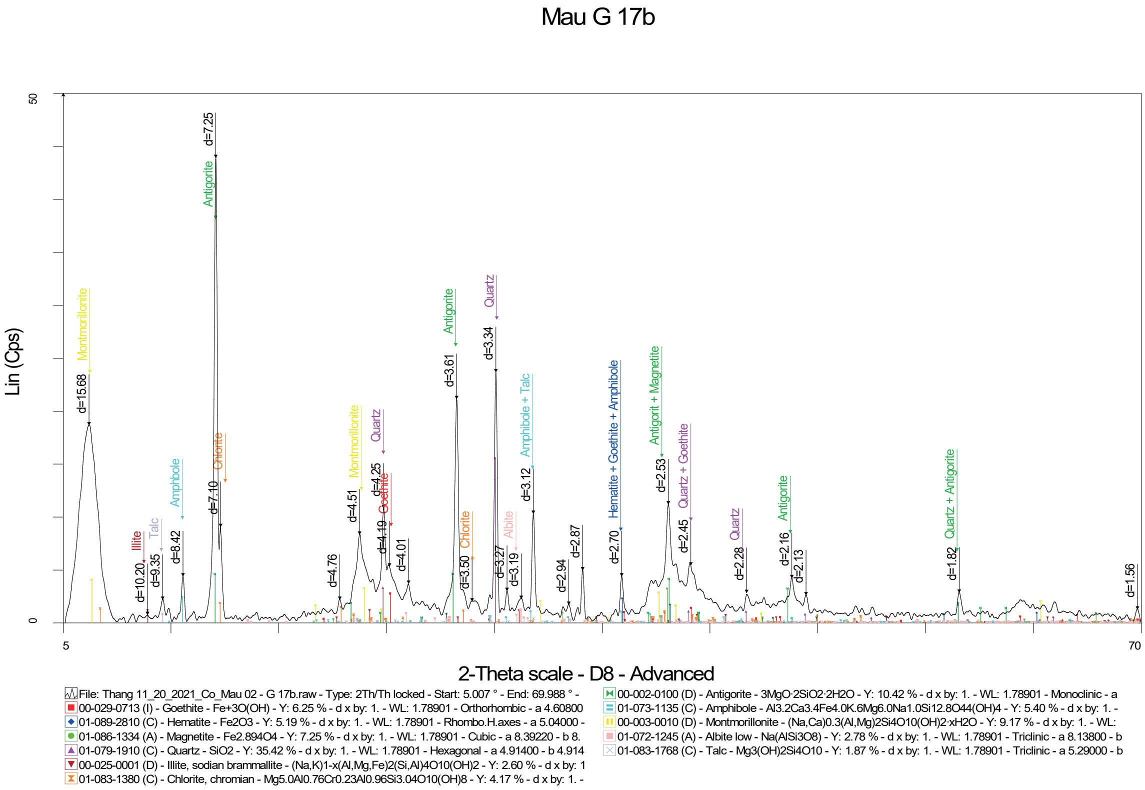


Fig. 4. *X-ray diffraction (XRD) pattern of a clay sized waste sample (G17b) from the Co Dinh area*

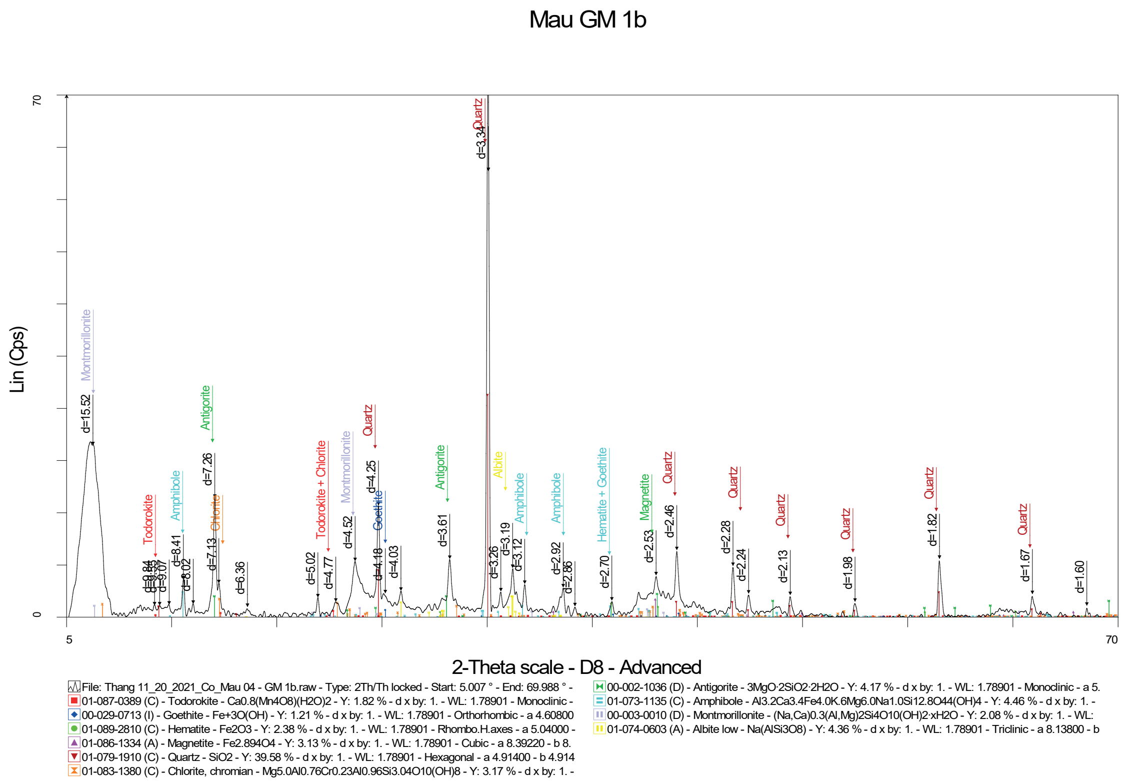


Fig. 5. *XRD pattern of a clay sized waste sample (GM1b) from the Mau Lam area*

Contents of Co and Ni

The analytical results of Ni, Co and some metals in clay-sized waste sludges from the Co Dinh and Mau Lam areas are shown in Table 3. The contents of Co (0.01–0.07%, an average of 0.05%) and Ni (0.35–0.72%, an average of 0.55%) in Co Dinh area are slightly lower than those at the Mau Lam area which has Co content varying 0.03–0.09%, an average of 0.07% and Ni 0.10–1.03%, an average of 0.78%. It indicates that there is a little difference in Co and Ni contents of the clay-sized waste in the Co Dinh and Mau Lam areas.

In comparison to the contents of Co and Ni in bedrocks and the weathering crust on the Nui Nua ultramafic massif (Table 4), it shows that Co and Ni are enriched in the weathering crust and waste sludges. However, the contents of Co and Ni in the clay-sized waste sludges are not much greater than those in the weathered serpentinites and other clayed zones in the weathering crust of the Nui Nua ultramafic massif.

Table 3
Analytical results of Co, Ni content and some metals in clay sized waste sludges

			Co Dinh area (34 samples)	**Mau Lam area (35 samples)**	**Combined area (69 samples)**
Content [%]	Co	max.	0.07	0.09	0.09
		min.	0.01	0.03	0.01
		ave.	0.05	0.07	0.06
		SD	0.01	0.02	0.02
	Ni	max.	0.72	1.03	0.09
		min.	0.35	0.10	0.01
		ave.	0.55	0.78	0.06
		SD	0.11	0.20	0.02
	Cr	max.	0.58	1.05	0.09
		min.	0.22	0.22	0.01
		ave.	0.30	0.53	0.06
		SD	0.07	0.22	0.02
	Fe (total)	max.	21.15	18.66	21.15
		min.	13.79	11.89	11.89
		ave.	16.09	16.41	16.25
		SD	1.90	1.53	1.73
	Mn	max.	0.65	3.24	3.24
		min.	0.08	0.21	0.08
		ave.	0.48	1.30	0.89
		SD	0.11	0.72	0.66

Table 4
Summary of Ni and Co contents in weathering crusts on the Nui Nua ultramafic massif

Sample ID	**Rock's description**	**Co [%]**	**Ni [%]**	**Sample location**
CD-07/2	greenish grey, moderately weathering serpentinites	0.020	0.33	car park next to Am Tien peak (Co Dinh area)
CD-10/1	greenish grey, weakly weathering serpentinites	0.014	0.24	serpentine mine (Co Dinh area)
CD-08/2	clay derived from brown and light green soft serpentinite (saprolite zone)	0.028	0.49	road to Am Tien Peak (Co Dinh area)
CD-16/1	light brown, moderately weathering serpentinite (saprolite zone)	0.025	0.53	foothill in Mau Lam area
CD-16/2	light brown, strongly weathering serpentinite, (saprolite zone)	0.024	0.47	foothill in Mau Lam area
Average		0.022	0.41	

Mineral and chemical composition in the debris waste sludges

The petrographic and mineralogical characteristics

The grain size distribution in the debris waste sludges indicates that the proportion of debris in the waste sludges from the Mau Lam and Co Dinh areas is quite similar, ranging 79–83%, with an average of 81%. The majority of the particles in the waste sludges range in size from larger than 2.5–0.074 mm.

The composition of the debris particles is highly diverse, including fragments of serpentinite, talc-altered dunite, ultramafic rocks, a few diabase fragments, chromite, pyroxene, plagioclase, quartz, and chalcedony mineral fragments, as well as spherical Fe-Mn nodules.

Table 5 shows the mineral compositions of the debris waste sludges from the Co Dinh and Mau Lam areas using differential thermal analysis, X-ray diffraction (XRD) and a Stereo microscope. It can be seen that todorokite and goethite minerals are consistently present in the samples, with significant contents. Figures 6 and 7 represent the XRD patterns of a debris waste sample taken from the Co Dinh and Mau Lam areas, respectively.

Table 5
Mineral compositions [%] of the debris sludge analyzed by XRD

Sample ID	**Mineral composition [%]**								
	todorokite	**goethite**	**hematite**	**magnetite**	**quartz**	**chlorite**	**antigorite**	**amphibole**	**nontronite**
Co Dinh area									
G6a	1–3	26–28	2–4	–	2–4	8–10	38–40	3–5	4–6
G12a	2–4	28–30	–	2–4	2–4	8–10	33–35	2–4	4–6
G15a	7–9	38–40	2–4	2–4	7–9	4–6	8–10	4–6	4–6
G18a	3–5	46–48	3–5	–	4–6	6–8	16–18	4–6	2–4
G20a	1–3	18–20	–	3–5	4–6	9–11	42–44	5–7	3–5
G23a	2–4	31–33	1–3	1–3	11–13	8–10	28–30	4–6	–
G28a	≤1	9–11	–	3–5	24–26	12–14	32–34	3–5	1–3
G31a	≤1	10–12	–	4–6	2–4	10–12	55–57	3–5	3–5
G41a	6–8	39–41	2–4	–	7–9	4–6	14–16	3–5	4–6
Mau Lam area									
GM2a	≤1	4–6	–	4–6	2–4	4–6	74–76	1–3	–
GM9a	8–10	20–22	–	–	7–9	6–8	18–20	8–10	4–6
GM12a	7–9	32–34	1–3	–	6–8	6–8	14–16	5–7	6–8
GM15a	1–3	22–24	1–3	3–5	1–3	12–14	34–36	3–5	4–6
GM17a	5–7	37–39	4–6	–	6–8	4–6	14–16	4–6	5–7t
GM25a	7–9	34–36	3–5	–	8–10	6–8	14–16	4–6	3–5
GM36a	7–9	41–43	4–6	–	8–10	4–6	7–9	3–5	3–5
GM41a	6–8	31–33	2–4	2–4	1–3	8–10	26–28	4–6	2–4
GM42a	16–18	24–26	–	–	12–14	4–6	6–8	4–6	3–5
GM43a	15–17	32–34	1–3	–	9–11	4–6	4–6	3–5	4–6

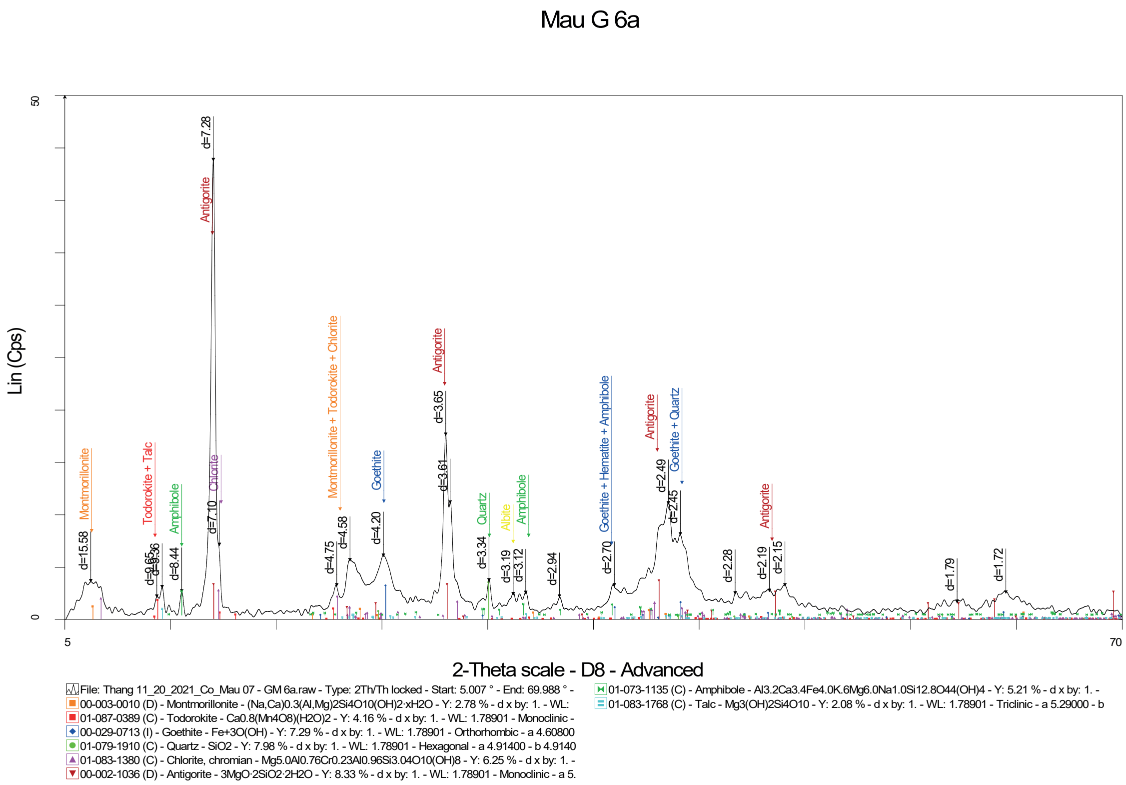


Fig. 6. *XRD pattern of debris waste sample (G6a) from the Co Dinh area*

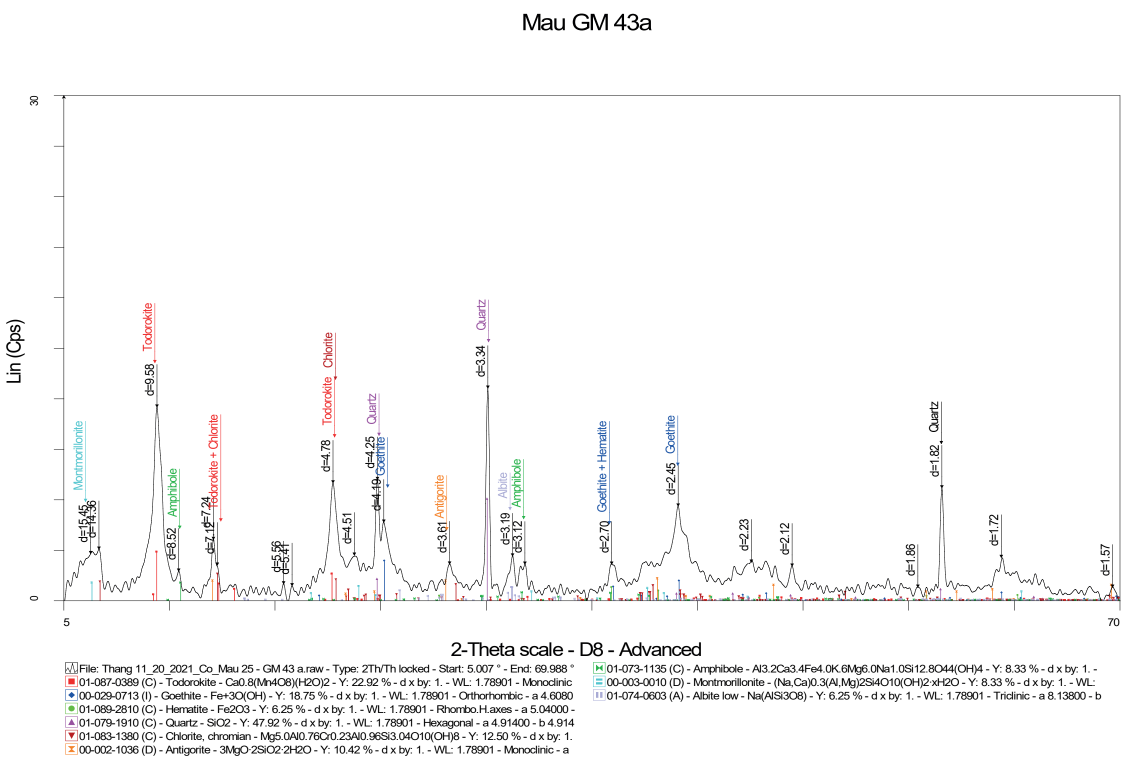


Fig. 7. *XRD pattern of debris waste sample (G43) from the Co Dinh area*

Characteristics of the Fe-Mn nodules

The Fe-Mn nodules can be visually recognized in the debris waste sludges. They are black, brownish black spherical or ellipsoidal in shape with smooth, shiny surfaces and irregular shapes, ranging in size from 1–2 mm to 7–8 mm (Figs. 8, 9). Morphologically, the Fe-Mn nodules in the debris waste sludges in the Co Dinh and Mau Lam areas bear resemblance to Mn nodules described in studies by Roqué-Rosell et al (2010), Gray and Eppinger (2012), Ugwu et al. (2019), Toro et al. (2020), and Tsune (2021).

The Fe-Mn nodules are often accompanied by rocks and mineral fragments within the debris waste sludges. In debris waste sludges in the Co Dinh and Mau Lam areas, the Fe-Mn nodules can constitute as much as 40–50% of the materials, covering the waste sludges with a dark appearance.

Under a polarizing petrographic microscope and reflected light microscope (Fig. 10), the Fe-Mn nodules consist of two components: serpentinite fragments and Fe-Mn cement, where the Fe-Mn cement acts as a binding material for the serpentinite fragments. The combination of these two components results in highly diverse structural morphologies, featuring zonal structures with concentric growth rings. Analysis reveals that the dark cement binding the serpentinite fragments in the Fe-Mn nodules consists of minerals such as todorokite, goethite, limonite and other iron hydroxide as cement, and manganese hydroxide (asbolane/psilomelane) as cement.

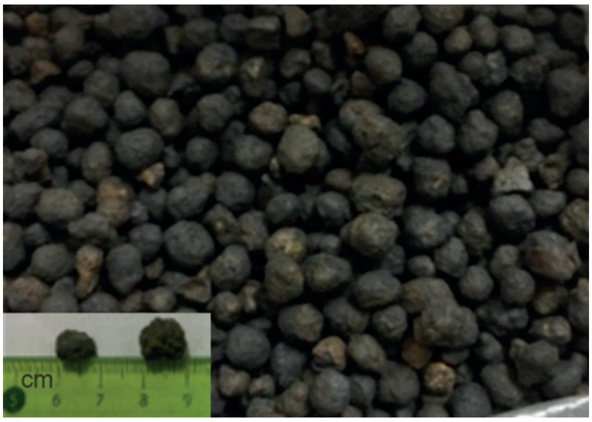


Fig. 8. *Fe-Mn nodules from Mau Lam area*

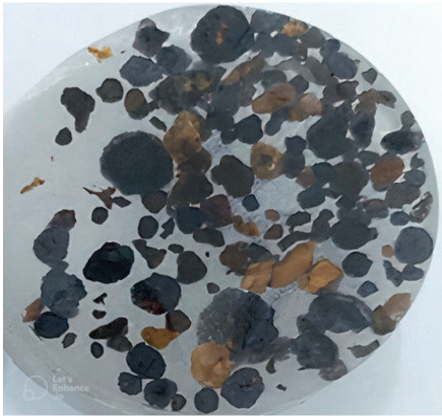
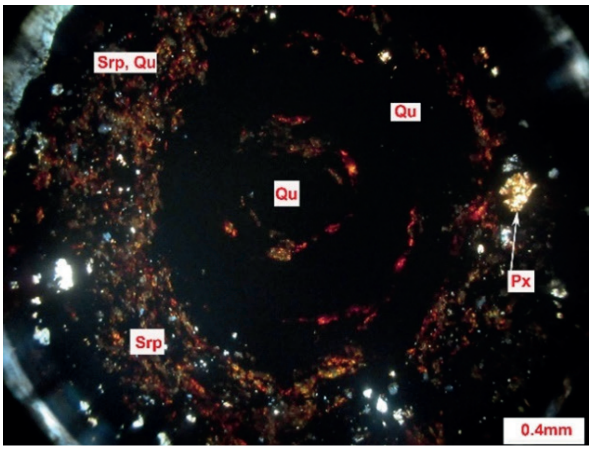


Fig. 9. *Rock fragments (mainly serpentinite and quartz) collected from debris waste sludges*



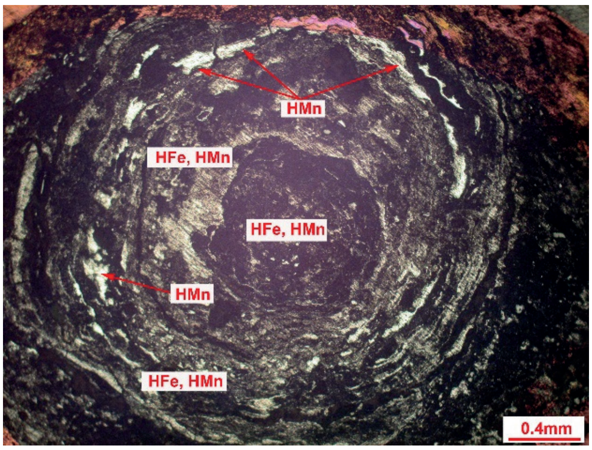
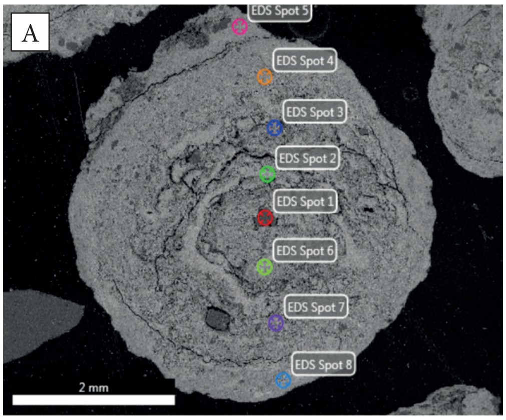


Fig. 10. *Black nodule in a sample (GM9) at of the debris waste sludge from the Mau Lam area: A) under polarizing petrographic microscope a combination of serpentinite (Srp) debris, quartz (Qu), and pyroxene (Px) (Nicol +) (A); B) under reflected light microscope: a combination of serpentinite (srp) debris and limonite (li), goethite (gh), manganese hydroxide (HMn) and iron hydroxide (HFe) colloidal matter*



B

Elements	Contents (%)							
	EDS Spot 1	EDS Spot 2	EDS Spot 3	EDS Spot 4	EDS Spot 5	EDS Spot6	EDS Spot 7	EDS Spot 8
Si	46.50	3.51	6.88	10.65	10.09	5.18	8.67	12.45
Al	0.26	1.27	1.37	0.51	1.43	2.00	1.99	2.09
Mg	0.03	1.45	1.93	0.23	0.52	3.42	2.90	1.67
Fe		8.10	12.83	43.84	46.6	9.48	12.22	34.26
Mn		44.62	36.23	3.46	0.61	38.78	33.31	6.83
Cr		0.15	0.36	0.65	0.52	0.17	0.17	0.57
Ni		5.86	6.41	0.76	0.55	9.81	5.33	1.03
Co		1.33	2.25	3.33	3.75	1.31	2.07	2.62

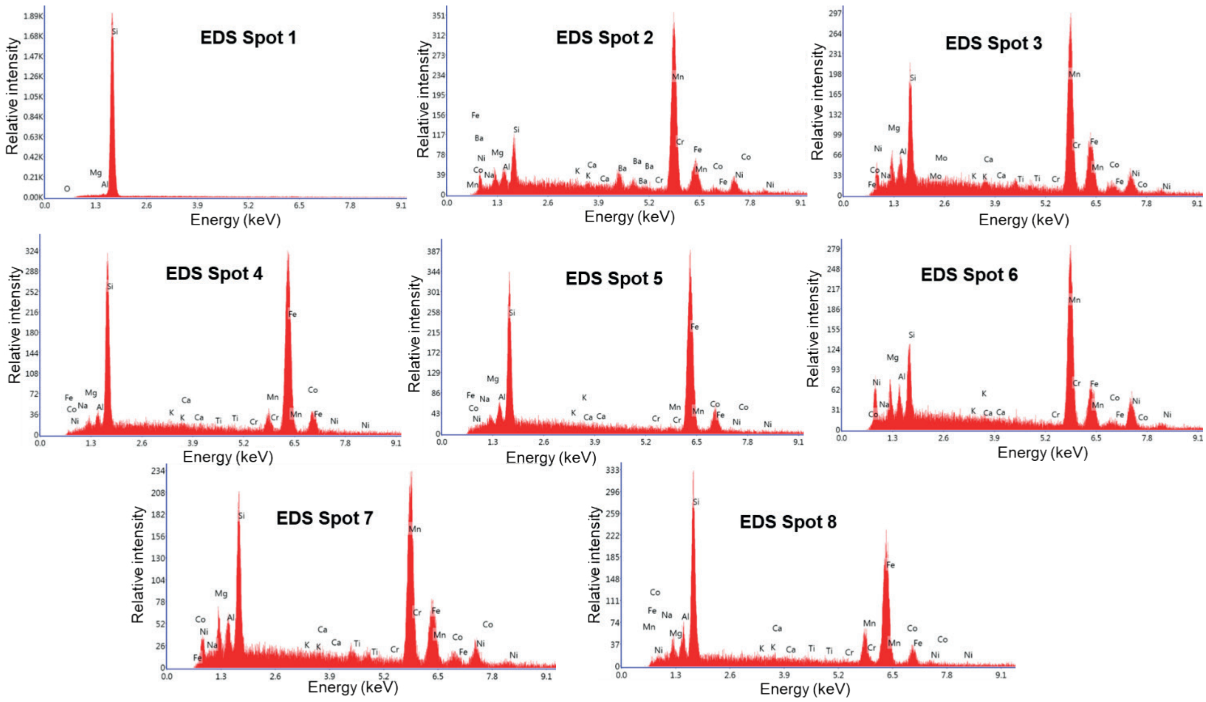


Fig. 11. *SEM/EDS analysis of the Fe-Mn nodule (sample GM9 in Mau Lam area): A) SEM image showing locations of EDS analysis spots; B) element contents at the EDS spots on the nodule; C) EDS patterns of the spots on the Fe-Mn nodule*

Figure 11 represents the SEM/EDS analysis of Fe-Mn nodule (GM9) taken in the waste sludges at Co Dinh area. The Co content at different spots on the Fe-Mn nodule varies relatively from 1.33 (Spot 2) to as high as 3.75% (Spot 5); while the Ni value relatively ranges from 0.76 (Spot 4) to as high as 9.81% (Spot 6).

Distributions of Co and Ni in the debris waste sludges

Table 6 summarizes the analytical results of the content of Ni, Co, Cr, Fe, and Mn in the debris waste sludges obtained from the Co Dinh and Mau Lam areas. As with the clay-sized waste sludges, contents of Co (0.03–0.38%, an average of 0.11%) and Ni (0.45–1.49%, an average of 0.78%) from the Co Dinh area are lower than those in the Mau Lam area which have Co (0.03–0.75%, an average of 0.34%) Ni (0.32–2.43%, an average of 1.63%).

It indicates that Co and Ni were enriched at the Mau Lam area compared with those in the Co Dinh area and the weathering crusts on the Nui Nua ultramafic massif.

Table 6
Analytical results of Co, Ni content [%] and some metals in debris waste sludges

Location	Content [%]					
	Statistics	Co	Ni	Cr	Fe (total)	Mn
Co Dinh area (40 samples)	max.	0.38	1.49	0.48	35.83	3.99
	min.	0.03	0.45	0.08	10.40	0.33
	av.	0.11	0.78	0.22	20.11	1.28
	SD	0.08	0.27	0.11	5.94	0.87
Mau Lam area (41 samples)	max.	0.75	2.43	1.14	57.79	11.00
	min.	0.03	0.32	0.13	7.76	0.29
	av.	0.34	1.63	0.47	25.05	4.68
	SD	0.15	0.45	0.22	7.37	2.31
Combined area (81 samples)	max.	0.75	2.43	1.14	57.79	11.00
	min.	0.03	0.32	0.08	7.76	0.29
	av.	0.20	1.12	0.33	21.94	2.65
	SD	0.17	0.56	0.21	7.11	2.44

Correlation between Co, Ni, and other associated metals

Figure 12 shows the correlations between the contents of Ni and Co, Ni + Co, and the total Fe + Mn (TFe + Mn) from the Co Dinh and Mau Lam areas. It can be seen that there is a direct relation between Ni and Co, between Ni + Co, and the total Fe and Mn in the debris waste sludges with high correlation coefficients. From the above data, there are the following remarks:

- The contents of Co and Ni in the debris waste sludges are significantly higher than those in the clay-sized waste sludges as well as in weathering crusts on the Nui Nua ultramafic massif. The clay-sized waste group has low contents of Co and Ni, making it impractical for the recovery of these metals.
- The debris waste group consists of rock fragments, mineral pieces, spherical dark Fe-Mn nodules rich in goethite, limonite, todorokite, and other Fe-Mn hydroxides. These nodules serve as the main carriers of Co and Ni, where Co and Ni are concentrated in Fe and Mn hydroxide minerals, such as todorokite, goethite, limonite, and asbolane (psilomelane).
- The Fe-Mn nodules in the debris waste sludges are more abundant in the Mau Lam area than in the Co Dinh area. The contents of Co and Ni in the debris waste sludges in Mau Lam are higher than those in the Co Dinh area.
- The debris waste sludges have a Co content of 0.11% and Ni content of 0.78% in the Co Dinh area, and Co of 0.34% and Ni of 1.63% in the Mau Lam area. These figures are significant compared to the standards set in the National Technical Regulation QCVN 49: 2012/BTNMT for geological and mineral mapping at a scale of 1 : 50,000 (2012), of which the minimum content of Ni in sulfide and in silicate ores is 0.5% and 1.0% respectively. The debris waste sludges in the Co Dinh and Mau Lam areas represent secondary Co and Ni ore bodies. This holds a practical significance for the extraction and recovery of Co and Ni.
- The direct correlation between Ni + Co with TFe + Mn and the high content of Ni and Co within the Fe-Mn-nodules suggest that in the debris waste sludges, Co, Ni are primarily concentrated within goethite, limonite, and other Fe-Mn hydroxide minerals. In these minerals, Ni and Co ions may substitute for Mn and Fe in the crystal lattice or in an adsorbed form (Tan 1956, Roqué-Rosell et al. 2010, Gray & Eppinger 2012, Ugwu et al. 2019).

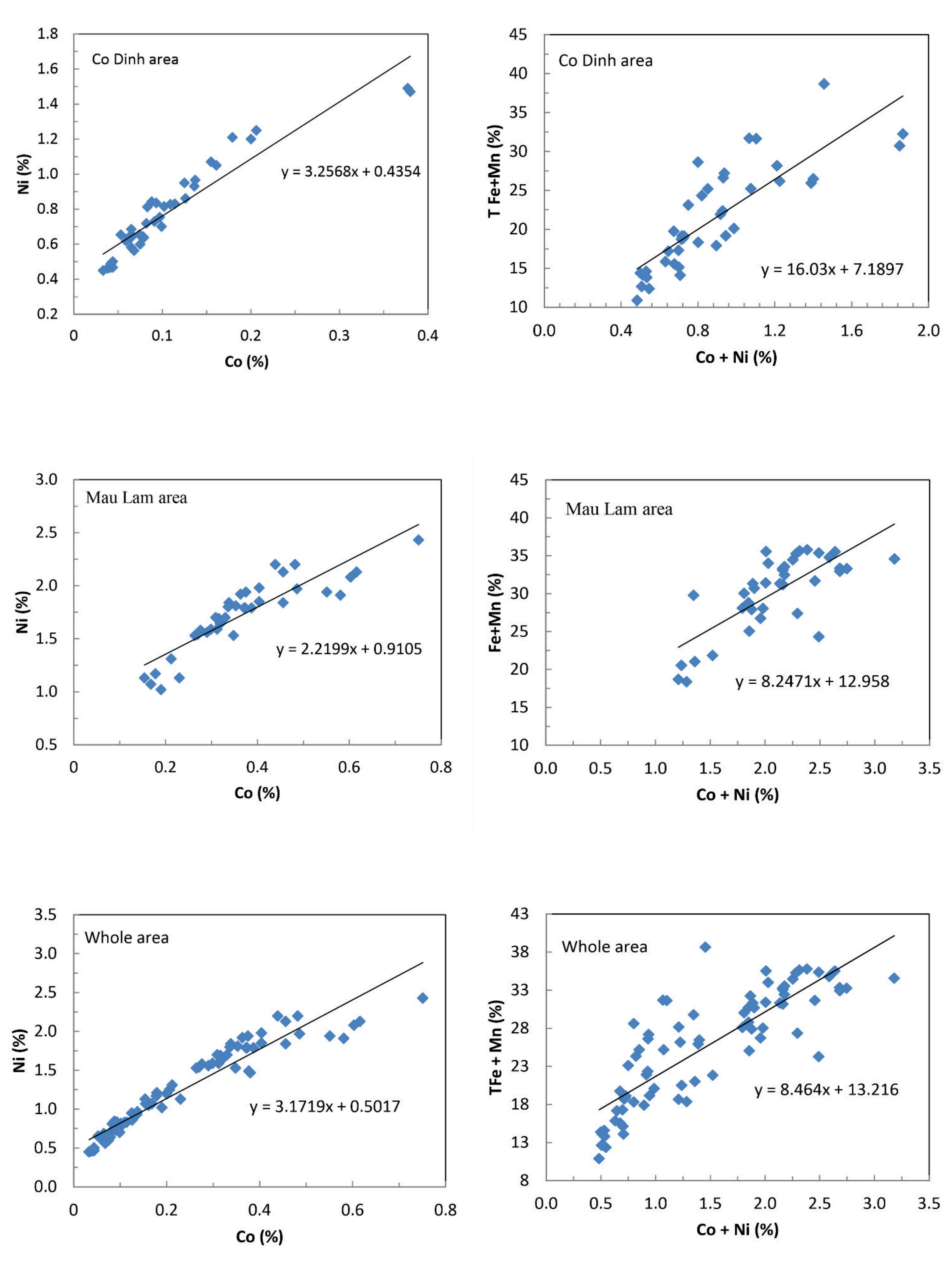


Fig. 12. *Graphs show the correlations between Co and Ni, between Ni + Co and Fe (total) and Mn minerals in the debris waste sludges (data from Table 6)*

Potential sources of Co and Ni in the explored chromite mining areas and in the debris waste sludges

As the clay sized waste group exhibits small contents of Co and Ni making it impractical for the recovery of these metals, estimation for potential sources of Co and Ni is therefore for the debris waste sludges, particularly with debris of grain size of 0.074–2.5 mm.

The estimation of potential sources of Co and Ni is based on data related to the published reserves of chromite ore and the distribution of Co and Ni in the debris waste sludges.

Dao (1996) reported that the contents of Co and Ni in the debris waste sludges are similar to those in granular particles in the original (unprocessed) ore bodies. The estimation of potential sources of Co and Ni is derived from the reserve estimation of chromite ores and the contents of Co and Ni analyzed samples in the debris waste sludges.

Potential sources of Co and Ni at Co Dinh area

According to Vietnam Geological Department (1963) and Dao (1996), the total estimated resources and reserves of chromite placer ore in Co Dinh area is 816,678,927 tons. According to Tuan (2012), in the period from 1966 to 2012, the total amount of raw chromite placer ores already exploited was 18,021,372 tons. Therefore, the remaining chromite placer ore is 798,657,555 tons (calculated by subtracting the exploited mount of 18,021,372 tons from the original reserves of 816,678,927 tons).

An amount of debris waste of at grain sizes ranging 0.074–2.5 mm which accounts for 33.2% (Table 1) in the remaining chromite placer ores is 265,154,308 tons (calculated by multiplying the remaining chromite placer ore of 798,657,555 tons by the content of the debris of grain size in the waste sludges 33.2%).

As with the averaged contents of Co of 0.11% and Ni of 0.78% for Co Dinh area (Table 6), the estimation of potential sources of Co and Ni metals in the debris waste sludges are 291,670 tons of Co (calculated by multiplying 265,154,308 tons by 0.11%), and 2,068,204 tons of Ni (multiplying 271,077,573 tons by 0.78%).

Potential sources of Co and Ni at Mau Lam area

According to Dao (1996), the total estimated resources and reserves of chromite placer ore in the Mau Lam area is 12,437,758 tons. According to Anh et al. (2014), the total amount of raw chromite placer ores already exploited was 158,181 tons. Therefore, the remaining chromite placer ore is 12,279,577 tons (calculated by subtracting the exploited amount of 158,181 tons from the original reserves of 12,437,758 tons)

An amount of debris waste of at grain size from 2.5 mm to 0.074 mm which accounts for 37.4% in the Mau Lam area (Table 1) in the remaining chromite placer ores is 4,592,562 tons (calculated by multiplying the remaining chromite placer ore of 12,279,577 tons by the content of the debris of grain size in the waste sludges 37.4%).

As with the averaged contents of Co of 0.34% and Ni of 1.63% for the Co Dinh area (Table 5), the estimation potential sources of Co and Ni metals in the debris waste sludges are 15,615 tons of Co (calculated by multiplying 4,592,562 tons by 0,34%), and 74,859 tons of Ni (calculated by multiplying 4,592,562 tons by 1.63%).

The total potential sources from the Co Dinh and Mau Lam areas are 307,284 tons Co and 2,143,062 tons Ni. In comparison with previous research, the total potential sources of Co and Ni as preliminary estimations are reasonable for the Co Dinh and Mau Lam areas. Dao (1996) reported that Co and Ni contents were recorded up to 0.05% and 0.7% respectively. Other works estimated total sources of about a quarter million tons of Co metals and 3 million tons of Ni. These numbers are in line with the paper's estimation.

The above estimation of potential sources of Co and Ni is preliminary and general, but reasonable and aligns closely with the reality as compared to resources of Co and Ni in the geological literature since the Nui Nua ultramafic massif is the greatest ultramafic in Vietnam. Apart from the Nui Nua massif area, Ni reserves recorded at 119,402 tons in Ban Phuc Nikel mine and 12,049 tons at Ta Khoa mine (Son La province), 52,548 tons at Suoi Cun mine and 81,000 tons at Ha Tri mine (Cao Bang province) (Tri et al. 2000). Lastly, the potential sources of Co in the Co Dinh and Mau Lam areas is regarded as unique in the mainland territory of Vietnam.

CONCLUSIONS

The waste sludges generated as a consequence of chromite mining are widely distributed in and around the Nui Nua ultramafic massif. The waste sludge in the Co Dinh area (northeast of the Nui Nua massif) is more abundant than that in the Mau Lam area (southwest of the Nui Nua massif). Two distinct groups of waste sludges are recorded: 1) clay sized wastes located in depressions and low-lying terrains; and 2) debris wastes distributed on relatively high lying terrain.

The clay sized waste group has low Ni and Co content, making it impractical for the recovery of these metals. The debris waste group consists of rock fragments, mineral pieces sourced from the Nui Nua ultramafic massif, spherical dark Fe-Mn nodules rich in goethite, limonite, todorokite, and other Fe-Mn hydroxides. These nodules serve as the primary carriers of Co and Ni. These two metals are concentrated within Fe-Mn hydroxide minerals, such as todorokite, goethite, limonite, and other Fe-Mn hydroxides (asbolane/psilomelane). They are components of the Fe-Mn nodules found in both the primary chromite placer orebodies and the debris waste sludges.

The debris waste sludges exhibit Co content of 0.11% and Ni of 0.78% in the Co Dinh area, and Co of 0.34% and Ni of 1.63% in the Mau Lam area. These figures are significant compared to the Vietnamese national quality standards set for the geological and mineral mapping, indicating that the debris waste sludges in these areas represent secondary Co and Ni ore bodies. This holds a practical implication for the extraction and recovery of Co and Ni in these waste materials.

The total potentially preliminarily estimated sources at and around Nui Nua massif are 313,800 tons Co and 2,189,264 tons Ni, of which the Co Dinh area has 298,185 tons of Co and 2,114,405 tons of Ni, and the Mau Lam area holds 15,615 tons of Co and 74,859 tons of Ni. These estimates are quite reasonable considering that the Nui Nua ultramafic massif is the largest ultramafic massif in Vietnam, and the Ni reserves at the study area aligns well with those recorded in the other parts of the country.

RECOMMENDATIONS

Further study is required to elucidate the distribution of Co and Ni in waste sludges as well as in the original ore bodies. Understanding the geological characteristics, conditions, and mechanisms of the formation of Ni and Co-containing Fe-Mn nodules is crucial. Examining the factors contributing to the variations in the content of these nodules, as well as Co and Ni contents between the Co Dinh and Mau Lam areas, will help to establish rational procedures for sampling and analyzing samples during the exploration and evaluation of Co and Ni resources in the original ore bodies.

It is essential to research effective beneficiation processes and recovery methods for Co and Ni in debris waste sludges. Strategies should be developed for the extraction and utilization of secondary Co and Ni ore sources at mining sites. Additionally, technological solutions and equipment for recovering debris wastes containing Co and Ni should be explored.

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