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## Clay Mineralogy of the Weathered Materials from the Serpentinized Peridotite in Minglanilla, Cebu Province, Philippines

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### Abstract

Serpentinized peridotite is an ultramafic rock that intrudes, during Cretaceous time, as widely scattered diapiric bodies along the principal fault zones of central Cebu, Philippines. This rock is exposed to chemical weathering in tropical conditions and through the leaching reactions leave a friable mantle of *in situ* regolithic materials. It is envisaged that the serpentine was derived from a pyroxene-rich peridotite.

The <2  $\mu$ m clay fraction of four regolithic materials obtained from this rockmass in Camp 7, Minglanilla were examined by means of x-ray diffractometry (XRD), differential thermal analysis (DTA), thermogravimetric analysis (TGA), scanning electron microscopy (SEM) and energy dispersive x-ray (EDX). Results of analyses showcase the polymineralic and heterogeneous nature of the weathered specimens with serpentine predominating, and could be differentiated possibly into two distinct assemblages. The first assemblage consists principally of serpentine-talc-chlorite-smectite and the second assemblage consists of serpentine-chlorite-smectite. Kaolinite is present in trace amounts in both suites. It is strongly perceived that these secondary minerals are genetic products from cadence of chemical alterations of primary ferromagnesian minerals particularly orthopyroxenes and clinopyroxenes.

The two assemblages appear to interpret the weathering reaction stages that the hypermelanic peridotite protolith had undergone. The first assemblage possibly corresponds to saprock (friable rock zone) which suggests the earlier stage and lesser intensity of weathering while the second assemblage possibly relates to saprolite (argillized solifluxed zone) which indicates the more advanced and greater intensity of weathering. The transition from the first assemblage to the second assemblage appears telescoping as indicated by the gradating loss of talc in the system. Both assemblages appear highly magnesian, ferruginous, siliceous and non-aluminous.

**Keywords** : Serpentine, chlorite, talc, smectite, saprock, saprolite, peridotite.

### Introduction

Santos-Ynigo (1951) envisaged that the serpentine exposed along the crest of the Toledo-Tabunoc road was evidently derived from a pyroxene-rich peridotite as suggested by the abundance of bastite pseudomorphs in thin sections made from that rock.

Aside from the mentioned bastite, no other secondary minerals were indicated and described in the existing literatures of BMG, 1981. It is apparent that the mineralogy and characteristics of the weathering products of the serpentinized peridotite is still ambiguous.

This research is therefore carried out with cardinal objectives to probe the mineralogy of the weathered

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regolithic materials obtained from the serpentinized peridotite by utilizing several analytical techniques available and to elucidate the dynamics of mineral formation and weathering.

## GEOLOGY

The following general description of serpentinized peridotite is lifted from Santos-Ynigo (1951).

Serpentinized Peridotite occurs mainly as widely scattered lenticular bodies along the principal fault zones of Central Cebu. It intrudes the Tunlob Schists and Pandan Series along tectonically weak axes of folding. Its close association with the Cansi Volcanics or with the Tuburan Limestone along fault zones probably indicates that it was injected along tight shear planes developed over wide area during a period of orogeny which followed the deposition of Tuburan Limestone. Renewed movements along the same planes of weakness at various times during the Tertiary brought the serpentine bodies up in contact with younger Tertiary formations.

## SAMPLES AND EXPERIMENTAL WORKS

Four samples (MAN-4, MAN-4A, MAN-5 and MAN-5A) of weathered materials on the surface contact of the serpentinized peridotite outcrop were collected from 2 sampling stations along Toledo-Tabunoc (Manipis) Road, Camp 7, Minglanilla, Cebu (Figure 1). The two stations are approximately 150-200 road meters distance apart. MAN-4 and MAN-4A are around 5 meters apart similarly for MAN-5 and MAN-5A. The weathered materials were obtained from friable outcrops and each sample was collected over an area of 1m<sup>2</sup> for each outcrop. These materials are regolithic in nature and are generally highly fragmented, porous, gritty and pulverable. The color is usually greenish gray.

Bulk samples for x-ray diffraction analysis were manually ground using agate mortar and pestle. The finely ground powders of each sample were mounted on glass slides by addition of few drops of distilled water, spread evenly by stainless spatula and allowed to dry naturally.

The <2 $\mu$ m size fraction of the regolithic samples were used for the various experiments. Fractionation into fine powders for multiple analyses was prepared following the recommended procedure of dispersion, sedimentation and centrifugation. X-ray diffraction (XRD) analyses of oriented <2 $\mu$ m size fraction samples mounted on glass slides by "natural setting method" were carried out using RINT 2000 x-ray diffractometer with a graphite monochromator (CuK $\alpha$  radiation) and operational setting of 30 kV and 14 mA. Differential thermal-thermogravimetric (DTA-TGA) analyses were made using Rigaku Thermo Plus TG 8120 DTA-TGA apparatus. Scanning electron microscopy (SEM) studies used Hitachi S-4000 FESEM. The specimens were coated with gold-palladium using FINE COAT ION SPUTTER JFC-1100. The same Hitachi S-4000 FESEM equipped with LINKS ISIS EDX operated at an accelerating voltage of 15 kV was utilized in conducting the energy dispersive x-ray (EDX).

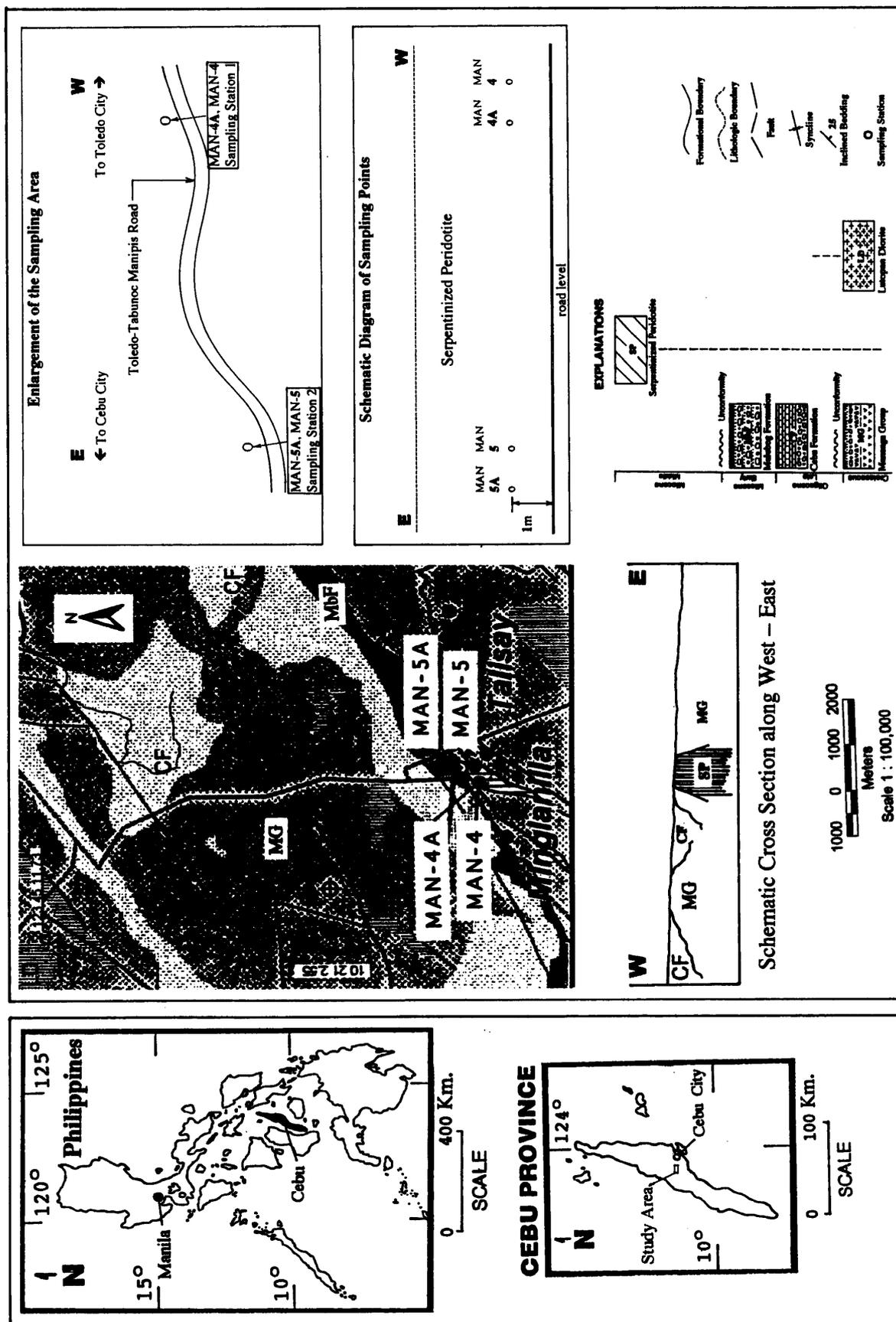


Figure 1. Geological and sampling location map of the study area in Minglanilla, Cebu Province Philippines. Based from the Philippine Bureau of Mines and Geosciences.

## RESULTS

## X-ray Diffraction (XRD)

X-ray diffraction diagrams of both bulk samples (Figure 2) and oriented fine ( $<2\mu\text{m}$  size) samples (Figure 3, top) disclose serpentine mineral as the main component of the weathered materials in the serpentinized peridotite. The whole assemblage is polymineralic, containing serpentine, chlorite, smectite, talc, and kaolinite. Serpentine is identified by the very strong and sharp peak reflections at  $\sim 7.3\text{\AA}$  and  $3.65\text{\AA}$ . Careful examination of the x-ray profile indicates that the serpentine is predominantly antigorite variety. Talc is revealed by series of strong and acute peaks at  $\sim 9.3\text{\AA}$ ,  $4.7\text{\AA}$  and  $3.1\text{\AA}$ . Moderately broad and high crested peaks at  $\sim 14\text{\AA}$ - $15.5\text{\AA}$  are superposed chlorite and smectite phases.

Ethylene glycolation (Figure 3, middle) shows the lability of smectite peak that swells to  $\sim 17\text{\AA}$  and separates with the stable  $14\text{\AA}$  peak of chlorite. Serpentine and talc are not affected by this treatment and maintained their respective peak positions as in the untreated state.

The 6N HCl treatment for 1hr obliterated all the peaks except talc, and the assemblages turned amorphous as illustrated by the featureless patterns (Figure 3, bottom). The talc peaks are very much enhanced here and they show well the great difference in abundance and the gradual disappearance from MAN-5, MAN-5A to MAN-4A, MAN-4. In this treatment that minimal presence of kaolinite is verified as indicated by the tiny peak trace at  $\sim 7.1\text{\AA}$ .

Series of heat treatment at 300, 500 and  $700^\circ\text{C}$  performed with moisture and EG treatments show the

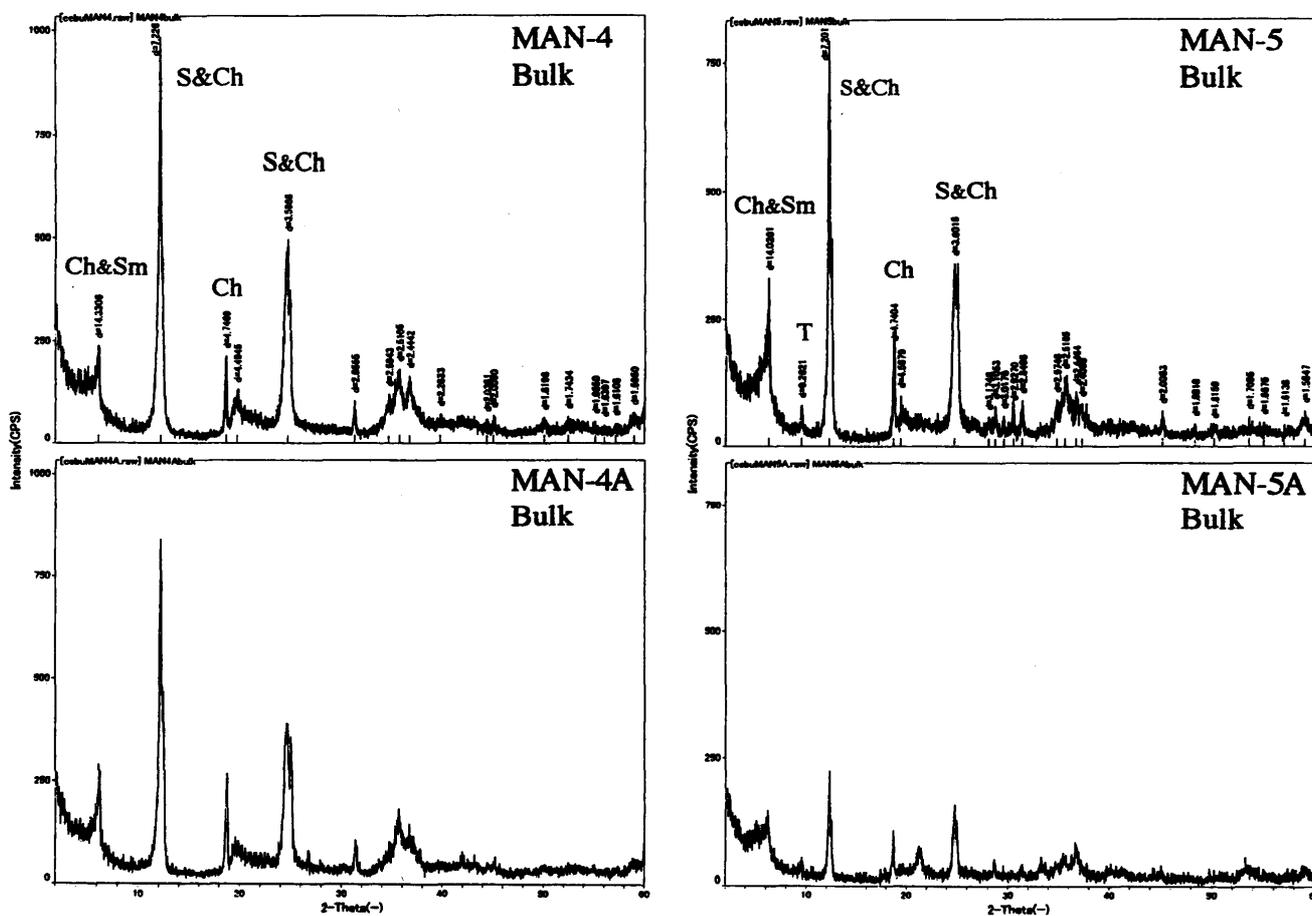


Figure 2. X-ray diffraction (XRD) patterns of the serpentinized peridotite bulk samples. S=serpentine, T=talc, Ch=chlorite, Sm=smectite.

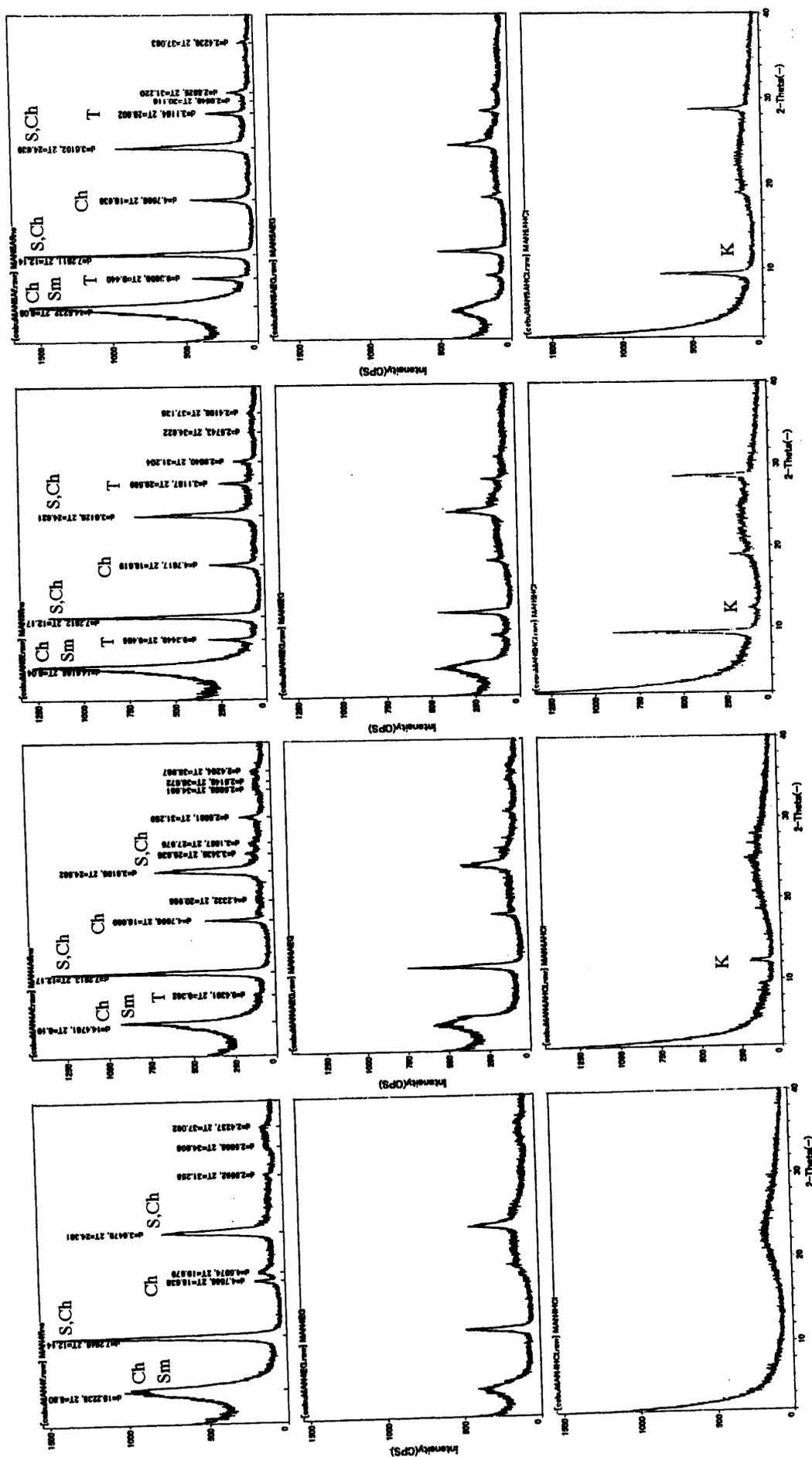


Figure 3. X-ray diffraction (XRD) patterns of the untreated (top), EG treated (middle) and HCl acid treated (bottom), parallel oriented <math>2\mu\text{m}</math> size fraction samples of the serpentinized peridotite. Left to right: MAN-4, MAN-4A, MAN-5 and MAN-5A.

contraction/expansion behavior of smectite and the inertness of the other phyllosilicates (Figure 4). The contraction to  $\sim 10\text{\AA}$  and expansion to  $\sim 15\text{\AA}$  (moisture) and  $\sim 17\text{\AA}$  (EG) of smectite may be observed up to

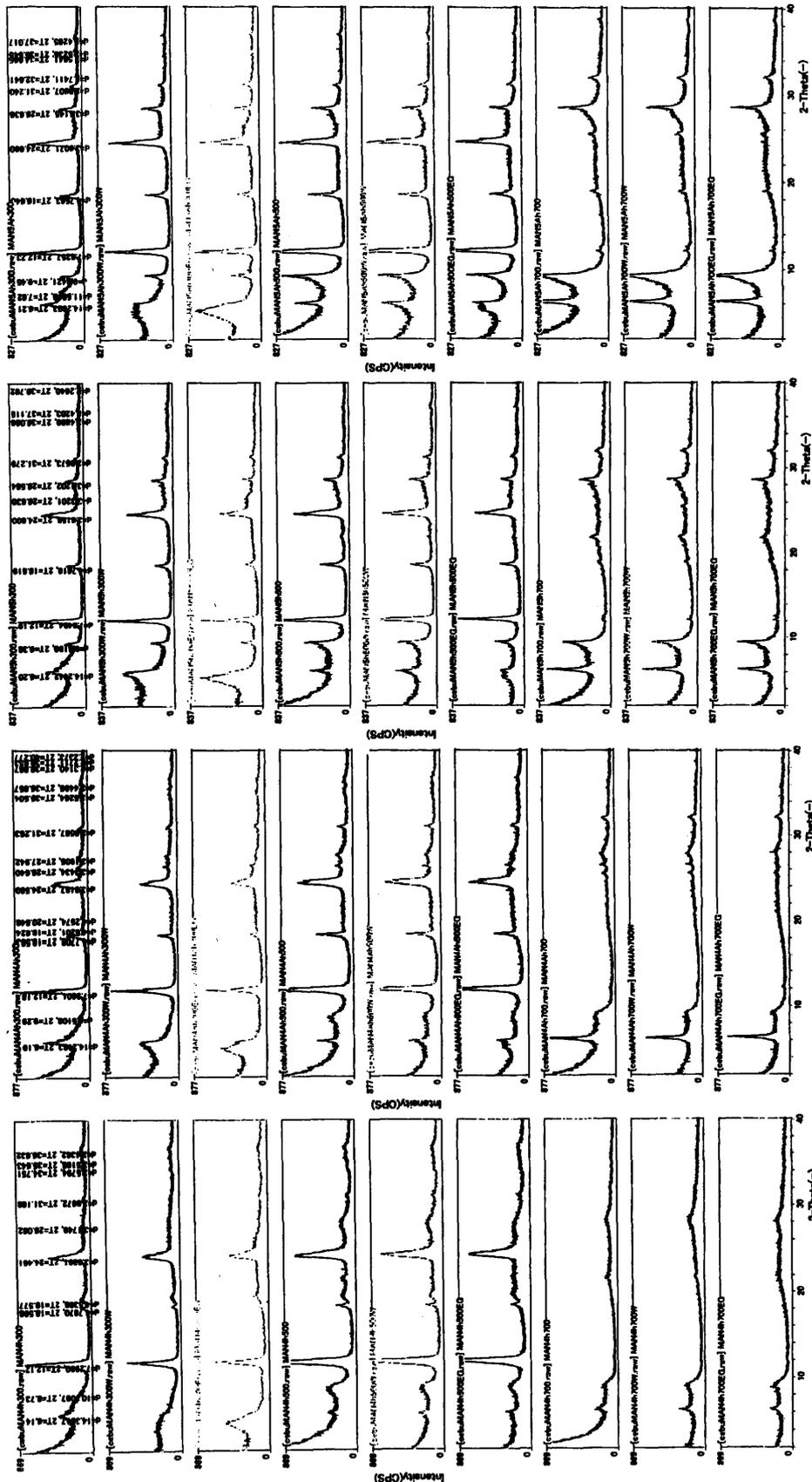


Figure 4. X-ray diffraction (XRD) patterns of  $<2\mu\text{m}$  size fraction samples of serpentinized peridotite heated at  $\{300^\circ\text{C}, 300^\circ\text{C} + \text{H}_2\text{O}, 300^\circ\text{C} + \text{EG}\}; \{500^\circ\text{C}, 500^\circ\text{C} + \text{H}_2\text{O}, 500^\circ\text{C} + \text{EG}\}; \{700^\circ\text{C}, 700^\circ\text{C} + \text{H}_2\text{O}, 700^\circ\text{C} + \text{EG}\}$ . Left to right: MAN-4, MAN-4A, MAN-5 and MAN-5A.

500°C in the 4 samples. This behavior ceases at 700°C where permanent contraction is attained as shown by the peak position at around 10 Å. The chlorite is greatly manifested by this heating process as shown by the rigidity of its peak position at ~14 Å and the greatest amplification at 700°C. Talc peaks also show gradual enhancement from 300°C to 700°C and this change is more observable in MAN-5 and MAN-5A. The serpentine peaks remain steady up to 500°C and deteriorate grossly at 700°C.

MAN-4 and MAN-4A subjected to 1200°C heat treatment for 1hr reverted to orthopyroxene, probably of hypersthene or bronzite variety (with traces of cristobalite) while MAN-5 and MAN-5A submitted to the same temperature level and heating period converted to clinopyroxene, possibly augite variety (with high amounts of cristobalite and tridymite) as shown in Figure 5.

### Differential Thermal Analysis (DTA)

The differential thermal analysis (DTA) curves are shown in Figure 6. The curves are generally characterized by 2 broad deep endothermic reactions. The lower endotherm lies in the <300°C range and the higher endotherm lies in the 500°C–600°C range. These endothermic and exothermic reactions are essentially superposition of reaction curves of all the minerals in the assemblages. The lower endothermic reaction corresponds to the removal of adsorbed and interlayer water from the smectite and adsorbed water from the other clay minerals. This reaction is generally displayed with peak positions at 77°C (MAN-4), 82.8°C (MAN-4A), 98.2°C (MAN-5), 92.4°C (MAN-5A)

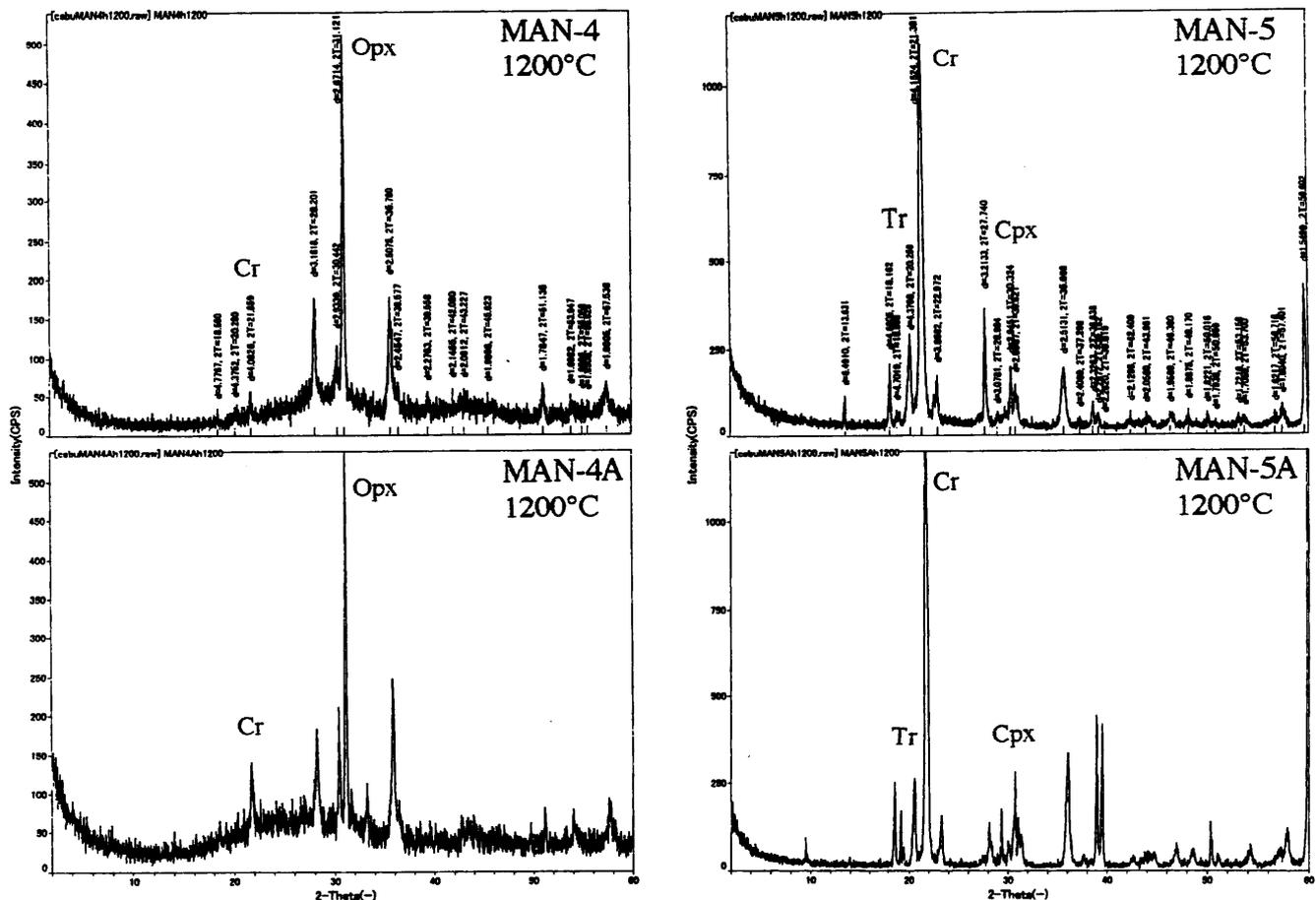


Figure 5. X-ray diffraction (XRD) patterns of <math><2\ \mu\text{m}</math> size fraction samples heated to 1200°C showing the high-temperature mineral products. Opx=orthopyroxene, Cpx=clinopyroxene, Cr=cristobalite, Tr=tridymite.

and shoulder at  $\sim 200^{\circ}\text{C}$  before reaching  $300^{\circ}\text{C}$ . The higher endothermic reaction corresponds to the dehydroxylation of all the clay minerals. This is most defined in MAN-4 with  $639.6^{\circ}\text{C}$  depression and slightly defined in MAN-4A, MAN-5 and MAN-5A with 2 irregular saddles. The prominent pointed exothermic reaction near  $800^{\circ}\text{C}$  may be attributed to serpentine and it suggests a clear phase change.

#### **Thermogravimetric Analysis (TGA)**

Abrupt weight loss in the range 7.55% - 11.11% is vivid before reaching  $200^{\circ}\text{C}$  and this is succeeded by gradual weight loss until  $1000^{\circ}\text{C}$  where total decrease in the range 18.45% - 21.87% is registered (Figure 6). The abrupt loss may be accounted for dehydration shown by all the samples. MAN-4 has another sudden weight drop of 7.52% in the  $600^{\circ}\text{C}$ - $800^{\circ}\text{C}$  coincident with dehydroxylation. The other samples only display continuous gradual drops in this OH expulsion regime.

#### **Scanning Electron Microscopy (SEM)**

Figure 7 shows the morphology of the 4 samples obtained by scanning electron microscopy (SEM). It may be observed that all the assemblages exhibit platy morphology and face-to-face aggregation. The platy morphology is virtually caused by the inherent platy and flaky character of all the phyllosilicates components. There is a discernible transition of morphology from densely packed flakes in MAN-5 (Figure 7 E & F) and MAN-5A (Figure 7 G & H) to shredded or stretched flakes resembling vermiform in MAN-4A (Figure 7 C & D) and MAN-4 (Figure 7 A & B) and the aggregation also becomes relatively porous. This change imparts evidence attributed to progressive weathering. Chrysotile serpentine is recognized by its tubular and splintery habit and is noted scattered very scarcely in the generally planar matrix of antigorite and other intimately associated phyllosilicates (Figure 7B).

#### **Energy Dispersive X-ray (EDX)**

The general chemistry of the two assemblages appear highly magnesian, ferruginous, siliceous and non-aluminous and does not show clearly a systematic trend of loss or gain of ions in the weathered profile (Figure 8). The high MgO (24.14%-27.43%) and FeO (12.28%-15.21%) and low  $\text{Al}_2\text{O}_3$  (2.74%-7.01%) may suggest that the octahedral sites of the phyllosilicates may be populated dominantly by Mg and Fe instead of Al. The high percentage of Fe also shows that oxidation potently took place.  $\text{SiO}_2$  ion is in the range 51.08%-56.61% which may be distributed to the tetrahedral position and discrete silica as well as amorphous impurities. Other elemental components are in insignificant amounts.

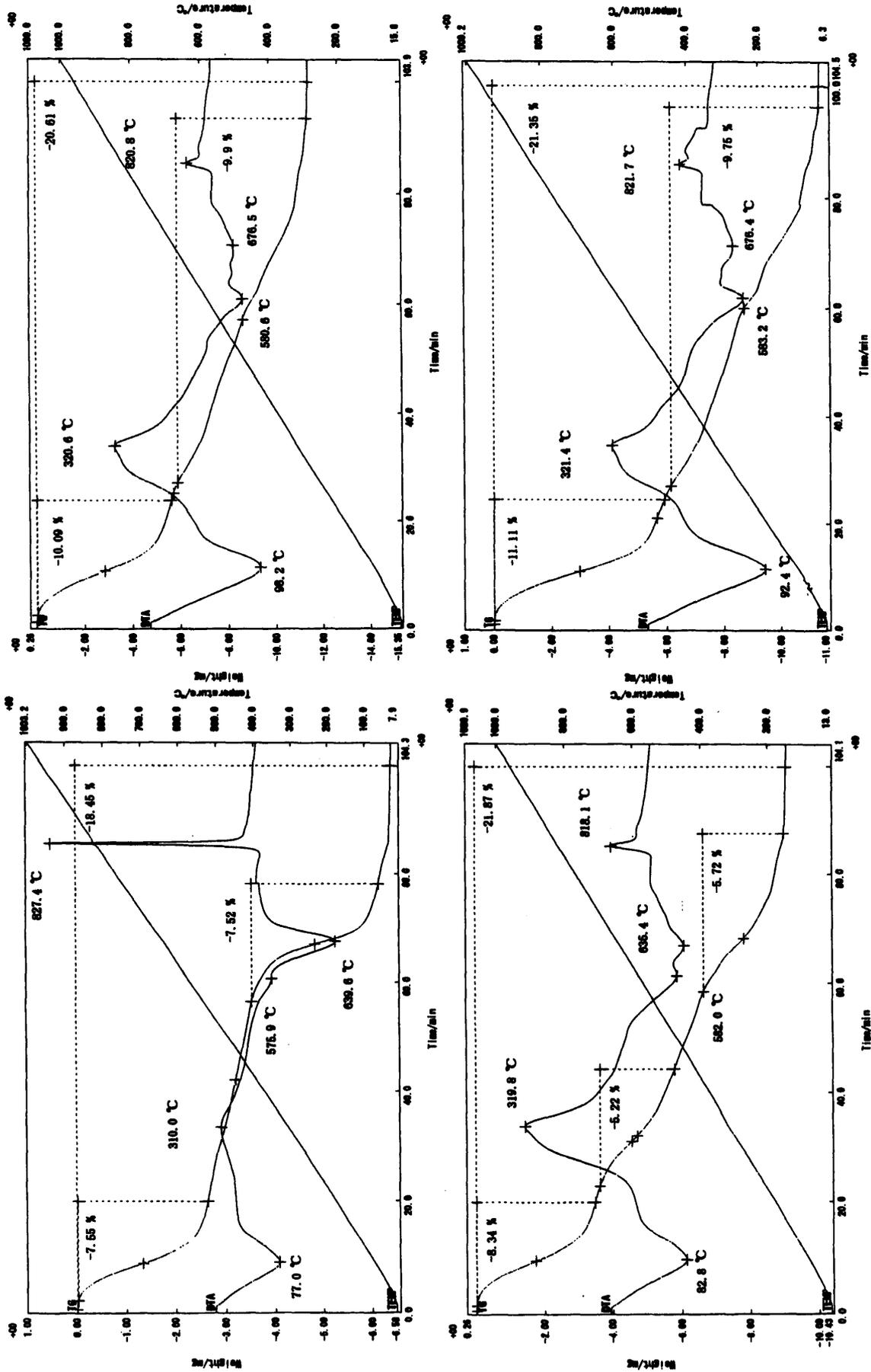


Figure 6. Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) curves of the 4 weathered serpentinized peridotite samples from Minglanilla, Cebu, Philippines.

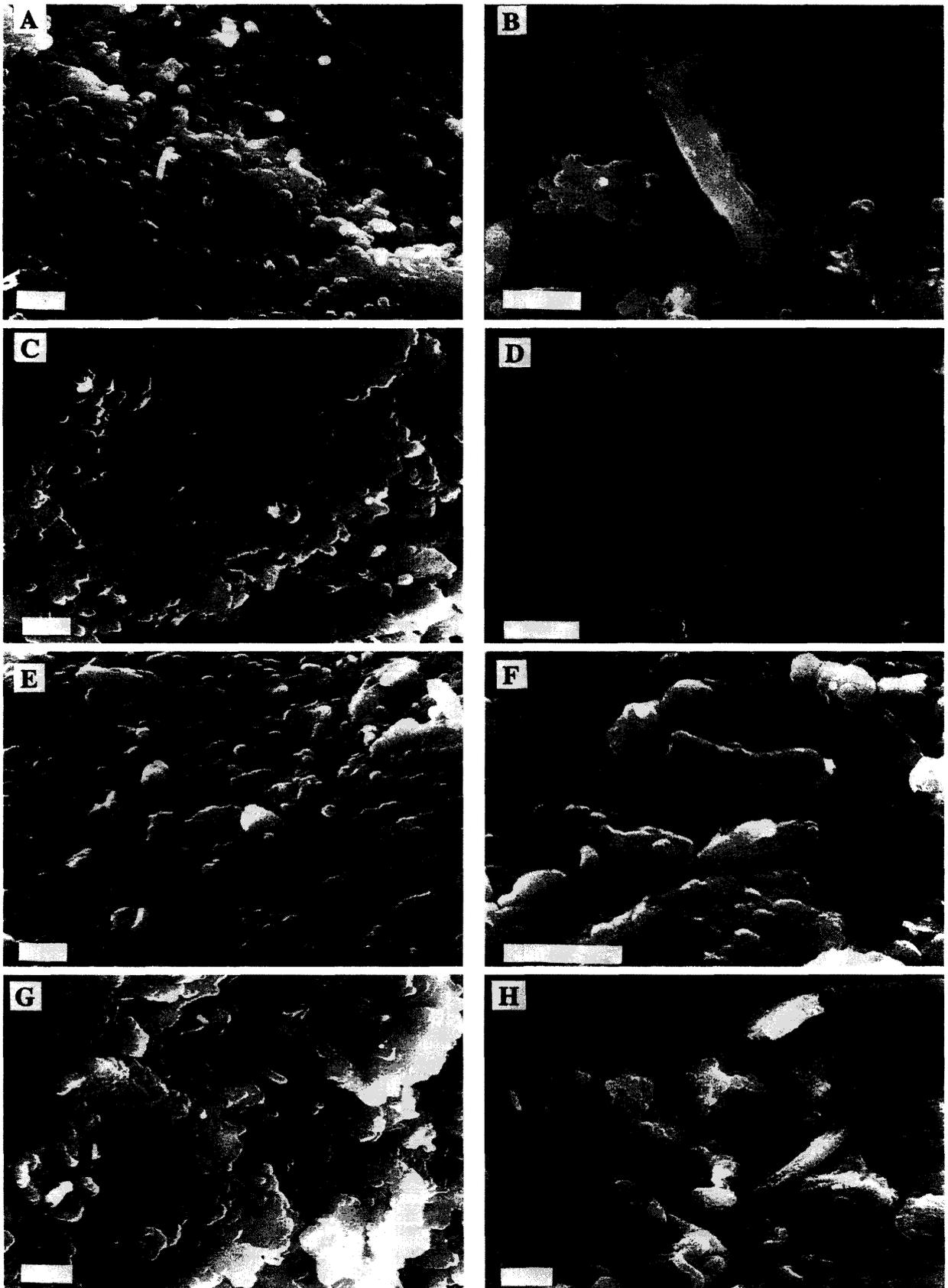


Figure 7. Scanning electron micrographs (SEM) of the 4 weathered serpentinized peridotite samples from Minglanilla, Cebu, Philippines. MAN-4=A&B, MAN-4A=C&D, MAN-5=E&F, MAN-5A=G&H. Bar scale=2microns

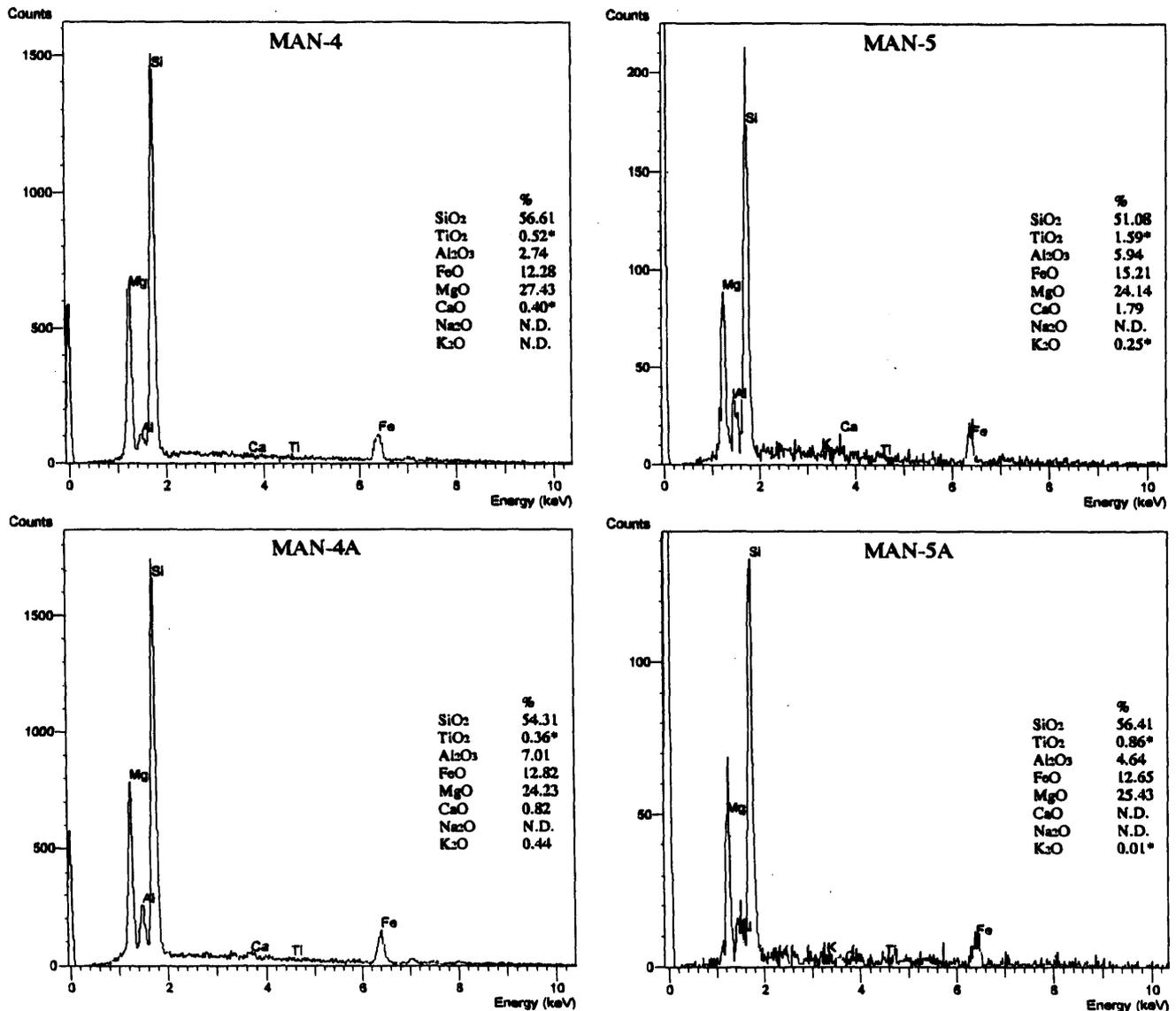


Figure 8. Energy dispersive x-ray (EDX) of the 4 weathered serpentinized peridotite samples from Minglanilla, Cebu, Philippines.

## DISCUSSIONS

Significant studies relating to the styles of weathering of ultramafic rocks were conducted by Fontanaud (1982), Ducloux et al., (1976), Rimsaite (1972), Eggleton and Boland (1982), Trescases (1979), Shirozu (1969), Shimane and Sudo (1958), Shirozu (1958), where they described the minerals and the sequential mineral alterations in the weathering zones.

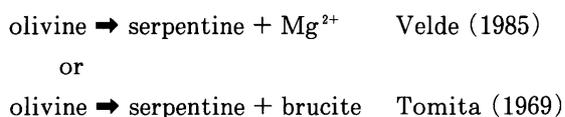
Ultramafic or ultrabasic rock weathers in different fashion than their mafic or felsic counterparts and the *in situ* regolith derivatives are quite different. Velde (1985) stated that the most striking features of the soil profiles developed on ultrabasic rocks are the very limited saprock zone and the rapid change from rock to clay mineral assemblages. The rock tends to weather as a whole and not in isolated chemical systems and this promotes

reactions between clay mineral species throughout a weathering horizon (Velde, 1985). He also emphasizes that the two factors which dominate the weathering of ultrabasic rocks are loss of magnesium from the system and oxidation of the iron which might be present.

The Serpentinized Peridotite in Minglanilla, weathers *en masse* in a very similar fashion described by Velde (1985). It lacks the grain contact reactions that live geometric network of pitted and etched surfaces on the parent rock that are typical features in felsic rocks. The regolithic materials on the weathering zone of the peridotite are generally thin, poorly developed and do not show distinct horizons even between the contact of the protolith and the regolith. Typically, the regolith is friable and loose and easily eroded. Field observations alone could not veritably distinguish the degree of weathering, the separation of saprock and saprolite zones, as well as identify and characterize the minerals present and their alteration sequence.

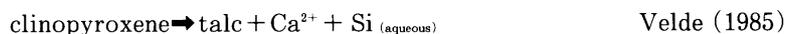
This ultrabasic type of weathering may also be compared to that of Righi and Meunier (1995) where they explained the weathering as a whole of serpentine and not in isolated reactions. According to Righi and Meunier (1995), the first reason is that the primary mineral size is frequently less to 1mm. The minerals are intimately mixed and at a small volume scale ( $\text{mm}^3$ ) the rock can be considered as homogeneous. Secondly, serpentine is a phyllosilicates bearing rock. Because of its homogeneity at small scale and high reactivity of magnesian phyllosilicates in surface condition, the rock appears to weather as a whole with no apparent microsystem effect at the thin section scales. Veblen and Busek (1980) and Eggleton and Boland (1982) demonstrated that olivine, pyroxene and amphiboles weather in a biopyroxene-sequence type. This type of weathering was not elucidated in the weathering of the peridotite in Minglanilla.

The serpentine-chlorite-talc-smectite assemblage shown by MAN-5 and MAN-5A, probably evinces the first weathering reaction of the hypermelanic peridotite protolith and thereby represents the initial phyllosilicates mineralogy conforming to saprock zone. The serpentine-chlorite-smectite assemblage shown by MAN-4 and MAN-4A, probably manifests the second weathering reaction and thereby represents the subsequent phyllosilicates mineralogy corresponding to saprolite zone. The removal of the talc from the second assemblage appears to proceed gradually as depicted by the diminishing intensity of talc-peak XRD reflections from sample MAN-4A to MAN-4 and this may suggest the lack of sharp boundary of mineral transition from the saprock zone to saprolite zone. These phyllosilicates were probably produced from destabilization of primary ferromagnesian, high-temperature minerals clinopyroxenes and orthopyroxenes. In the absence of primary mineral phases obtained from the protolith, this deduction is based on the ferromagnesian minerals produced from the thermal treatment at  $1200^\circ\text{C}$ , which yielded clinopyroxenes from MAN-5 and MAN-5A, and orthopyroxenes from MAN-4 and MAN-4A, shown in the XRD patterns (Figure 5). The said mineral products obtained at  $1200^\circ\text{C}$  thermal treatment in this study are quite different from the ones cited by Nemeč (1981) and Tomita (1969) in their mineral identification methods where forsterite is the principal product obtained at same temperature treatment. It appears in this study that pyroxene minerals may be dominant than olivine in the parent rock which is in agreement to observation of Santos-Ynigo (1951). However, since peridotite is the chief rock, it is certain that it also contains olivine in appreciable amounts. And according to Velde (1985) serpentine forms mostly from olivine but not in the presence of silica as seen in the reaction:

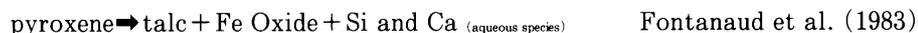


This serpentinization of olivine is promoted under alkaline condition and the reaction may even proceed at lower temperature or in a shorter period (Tomita, 1969) than the case reported by Bowen and Tuttle (1949).

Talc is a highly siliceous low temperature phase, as would be expected from the reaction:



or



The highly siliceous talc associated in MAN-5 and MAN-5A might be inferred as vital contributor of some of the  $\text{Si}^{4+}$  in cristobalite and tridymite formation in the 1200°C fired product. The seeming loss of talc in MAN-4 and its trivial amount in MAN-4A appears to show the poorer inclusion of cristobalite and tridymite in the 1200°C fired product in contrast to the former 2 (Figure 5).

The serpentine is dominated by antigorite variety, however, sporadic association of chrysotile is also observed. It is not clear at present whether the smectite that formed first was saponite (Mg-rich) in the saprock and gradually transformed to nontronite (Fe-rich) in the saprolite. Righi and Meunier (1995), however, exemplified that orthopyroxene and clinopyroxene in the alteroplasma microsystem (saprock) and in the pedoplasma microsystem (saprolite) would generally weather to Fe-saponite following the pathway  $\text{Opx} + \text{Cpx} \rightarrow \text{Fe-Sap}$ . It may be inferred, however, that the smectite in the four samples are all Mg- and Fe-rich since they all show vulnerability to HCl acid treatment based on the disappearance of their peak reflections in the XRD patterns. Chlorite may be the usual Mg-rich type on the basis of their x-ray peak reflections. Kaolinite is associated in negligible amounts in both assemblages. Quartz and possibly amorphous silica are scarce accessories.

The important parameters which control the mineral reactions occurring during weathering of ultrabasic rocks are chemical potential of Mg ( $\mu_{\text{Mg}}$ ) and the oxidation of  $\text{Fe}^{2+}$  ions (Righi and Meunier, 1995; Velde, 1985). The chemistry of all four Minglanilla samples is highly magnesian, ferruginous, siliceous and non-aluminous and apparently no distinct pattern of loss or gain of ions may be discerned and therefore the loss or diminution of Mg and oxidation of Fe are not explicit. Righi and Meunier (1995) cited however that the individual effects are difficult to distinguish since the decreasing of  $\mu_{\text{Mg}}$  is concomitant with the oxidation increase during the weathering process. These parameters are independent:  $\mu_{\text{Mg}}$  is an intensive variable whereas the oxidation modifies the  $\text{Fe}^{3+}$  composition of the microsystems (inert components) (Righi and Meunier, 1995).

## CONCLUSIONS

The weathered regolithic materials derived from the serpentinized peridotite are essentially polyminerally, with the dominance of mineral serpentine in the bulk and fractionated specimens. Other essential phyllosilicate minerals are chlorite, talc, and smectite. Kaolinite is associated in extremely subordinate amounts. Quartz impurities are sporadic. The minerals are highly presumed to be derivatives of sequential chemical alterations of ferromagnesians chiefly orthopyroxenes (hypersthene or bronzite) for MAN-4 and MAN-4A, and clinopyroxenes (augite) for MAN-5 and MAN-5A. The serpentine-talc-chlorite-smectite and serpentine-chlorite-smectite are the 2 significant clay mineral emblems differentiated that reflect weathering stages and intensity in the hypermelanic peridotite protolith. The first assemblage presumably links to saprock zone and the second one to saprolite zone. The saprock to saprolite transition is not distinct but gradual, gauged from the mineral associations that indicate the progressive evacuation of talc from the system. Likewise, the transition is apparently not accompanied by distinct trend of changes in the bulk chemical compositions. Morphology change, however, shows gradual transition from dense flakes to shredded flakes.

The bastite pseudomorphs were not identified in this study but the pyroxene-rich nature of the peridotite is confirmed.

## ACKNOWLEDGEMENT

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