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# X- RAY DIFFRACTION AND MINERALOGICAL STUDY OF VERTISOL IN EASTERN MACEDONIA

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#### ABSTRACT

The paper is about mineralogy of vertisols spread out in Eastern Macedonia. Three basic soil profiles were selected to present the vertisols that occur in different parent materials (tertiary clay sediment, andesite tuff and andesite brecca).

X-ray diffraction (XRD) is known as the best method for the identification and quantification of minerals present in soil. This method is used in our research for determination of present minerals. The XRD results indicate that the main constituents of the coarser soil fractions (silt and sand) are quartz, feldspates and calcite, while in the profile developed on andesite breccia two very rare minerals were determined (Koashvite in hor. Aca is present with 81% and Winchinite in hor. C is present with 45.60%). In the finest clay fraction, montmorillonite and mixture-layer minerals (MLM) are dominant in all cases.

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**KEY WORDS** 

X-ray diffraction; clay minerals; koashvite; winchinite.

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#### [I] INTRODUCTION

In Macedonia, the vertisol is spreading in about 61.900 ha. It is present mostly in the valleys and melioration areas (Stip, Probistip, Zletovo, Ovce Pole, Kumanovo, Skopje, Malesh and Pelagonia). Vertisols have high potential for the production of different food crops (wheat, maize, barley, garden-stuff, fodders, vine yards).

This type of soil is not much explored especially in the region of East Macedonia, as in physical and chemical characteristics, and in its mineralogy. In the coarser mineralogy fractions of vertislol there is a dominance of feldspate (usually 50-70%), and of quartz (20-30%) [1]. In the vertisols of Malesh and Kumanovo the montmoriilonite dominates in clay fraction [2]. There are similar results for the vertisols from Ghana, Congo, India, Morocco and Nigeria [3]. The dominance of montmorilonite in clay fractions is confirmed by numerous researchers from neighbouring Serbia [4, 5], Bulgaria [6, 7, 8] and Albania [9].

Subject of this paper is the mineralogy of vertisol in central Macedonia (Stip and Probistip). The method which is used in laboratory work for mineralogy research is roentgen difractometric method.

### [II] MATERIALS AND METHODS

The Three sites were selected to present vertisols occurring in different parent materials of the study area: profile  $N^{\circ} 2$  – on clay sediment (Stip -

41°49'06.42" N, 22°11'59.71" E); profile N° 6 – on andesite tuff (Probistip - 41°54'20.74" N, 22° 09'47.95" E); and profile N° 8 – on andesite breccia (Probistip - 41°53'34.76" N, 22°11'05.64" E). Thirteen soil samples were collected from the Ap (cultivated layer of humus-accumulative horizon), A (non-cultivated layer of humus-accumulative horizon), AC (intermediate horizon) and C (parent material) horizons of pedons for laboratory analysis.

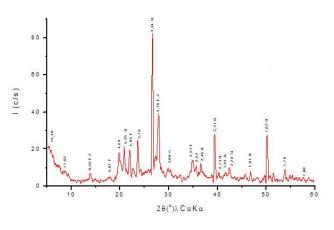
All soil samples were crushed and separated. From the individual samples were taken bigger particles and examined under binoculars. Semiquantitative analyses were performed on all soil samples. The proportions of minerals in the samples are calculated under the ratio of reflections' intensity with the greatest intensity.

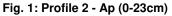
The samples were shot by Difraktometar type PHILIPS PW 1051 in the area  $2\theta = 50 \div 600$  for determination of the mineralogy composition without making clay analysis ("general difraktograms"). Copper radiation with a wavelength of  $\lambda$  CuK $\alpha$  = 1.54178 Å was used. The voltage on anticathode of generator "NORELCO" was 40 kV, and electric current 30 mA. The counter was moving at a speed of  $2\theta = 20/\text{min}$ . For determination of clay minerals from each sample were made two preparations in which one was shot as untreated, then saturated with glycerine and the other was burning at a temperature of 480 °C to determine the type of clay minerals in the area  $2\theta = 3^0 \div 14^0$ . This means that each sample is recorded four times (once in the entire region  $2\theta = 5^{\circ}$ ÷ 60°) and three times: untreated - N, saturated with glycerine - G and burning at 480 °C - Z [9, 10, 11, 12]. For identification of minerals JCPDS (ASTM) file was used. Very weak reflections were not taken into accounts which are insignificant for the identification of minerals or reflections that could not be identified on this ways.

CHEMISTRY

## [III] RESULTS AND DISCUSSION

Hor. Ap (0-23cm): The sample contains quartz (Q - 72.60%), feldspates (F - 15.40%), clay minerals - montmorillonite (M -6.80%). Calcite (C) is present with 5.20% and reflections that indicate that the zeolite is present in very small quantities are noticed [Figure-1].





Hor. A (23-75cm): This sample also contains quartz at most (Q -60.51%), then clay MLM minerals (M - 13.75%), feldspates (F) and calcite (C) 10.70%, and ilite-mica are present with 5.50% [Figure-2].

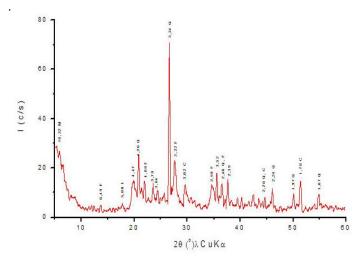
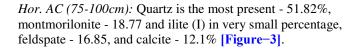


Fig. 2: Profile 2 - A (23-75cm)



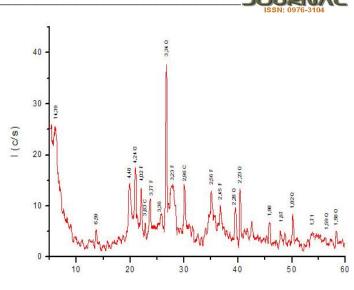


Fig. 3: Profile 2 - AC (75-100cm)

Hor. C (100-120 cm): Parent material differentiates from the other above mentioned horizons in a way that the reflections from calcite (C - 32.00%) and guartz (Q - 36.00%) have almost equal intensity. Feldspate reflections are 13.70%, clay minerals -12.60% and the minerals from ilite-mica group - 5.30%. As for the clay minerals, they are very mixture-layered types in which montmorilonite layers participate and there is a negligible quantity of chlorite [Figure-4].

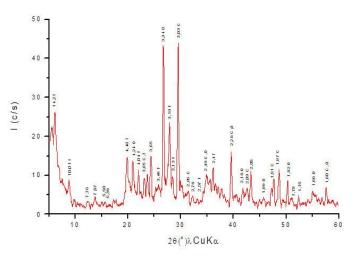
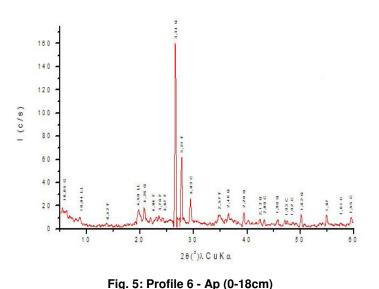


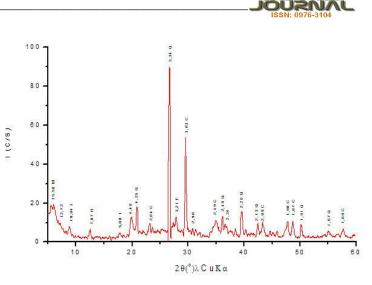
Fig. 4: Profile 2 – C (100-120cm)

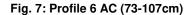
Hor. Ap (0-18 cm): As before, in this sample the most present is quartz (Q - 61.10%), then feldspate (F - 26.00%), calcite (C -6.10%), clay minerals (M - 3.50%) are mixture-layered with layers of montmorrilonite mixed with ilite-mica layers even though there are free ilite-mica minerals ((I, L) - 3.30 %). Reflections of hlorite in very small quantities are noticed [Figure-5].

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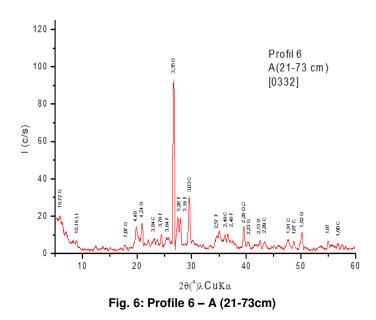




*Hor.* A (21-73 cm): The sample contains mostly quartz (Q – 62.40%). Calcite is present with 18.40% and feldspate is present with 8.00%. Clay minerals are consisting mainly of mixed-layers of montmorillonite, ilitte and chlorite are present with 6.70%, while free mica-ilite minerals are present by 4.50% [Figure-6].

*Hor.* C (107-130cm): this sample contains mostly quartz (Q - 46.20%), calcite (C -28.10%), than mixture-layered silicates (montmorillonite-chlorite) with 8.20%. Feldspate, dolomite and ilite-mica are present with 5.80% [Figure-8].

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*Hor. AC (73-107cm):* Dominant mineral is quartz with 67.80%. Calcite is presented with 23.00%. Clay minerals are presented with 4.80% (mainly ilite-montmorillonite). Free ilite-mica minerals are present by 4.00%. The presence of chlorite in very small amounts is also noticed [Figure-7].

*Hor. Ap* (0-26cm): Feldspate is mostly present (F-56.10%) and then quartz (Q -36.40%). Clay minerals are present with 7.4%. Ilite and calcite are present in small amounts [Figure-9].



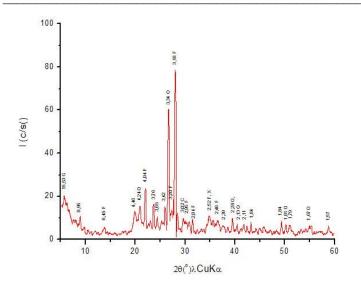
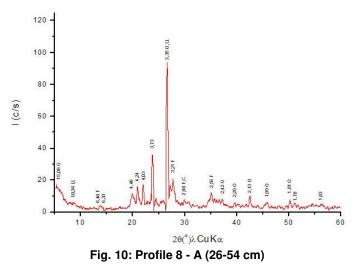


Fig. 9: Profile 8 - Ap (0-26 cm)

*Hor.* A (26-54 cm): in this sample dominant mineral is quartz with 75.10%, then feldspate (F - 11, 6%). Clay minerals are present with 5.30% (ilite-montmorilonite and ilite-chlorite). Calcite has approximately the same 5.30%, while free ilite is present with an insignificant quantity [Figure-10].



*Hor. Aca* (54-105 cm): The most common mineral in this sample is very unusual. Attempts by the program "MPDS" indicated a very rare mineral Koashvite Na<sub>6</sub> (CaMn)(Ti,Fe)SI6O18 which is registered in the records JCPDS (ASTM) under N°. 27-669. By reflection intensity of 2.52 Å, this mineral (K) is present around 81% and then quartz 12.30%. Feldspate are mostly from albeit type with 4.7%, clay minerals is present with 2.0% and they are consisted of swelling components. Reflections of free ilite are also noticed [**Figure-11**].

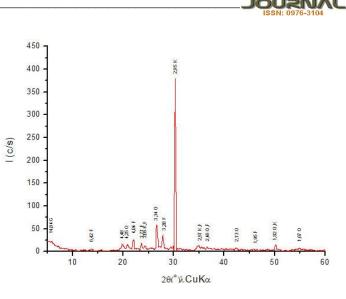


Fig. 11: profile 8 - Aca (54-105 cm)

*Hor. AC (105-140 cm):* the most common mineral is quartz (Q - 48.50%), than feldspate (F – 17.00%). Significant is the presence of cordierite (17.00%). Clay minerals are present with 7.50% and these are mainly mixed-layered minerals ilite-montmorillonite (ilite-chlorite) and pure chlorite. Minerals from ilite-mica group are present with 4.00% [Figure-12].

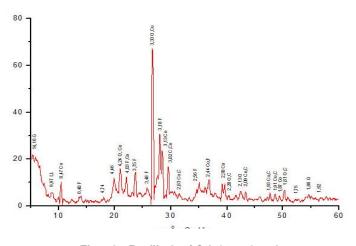


Fig. 12: Profile 8 - AC (105-140 cm)

Hor. C (140-155 cm): in this sample the most present mineral was difficult to determine. According to the literature review, by using the program "MPDS and JCPDS (ASTM) files, reflections winchite correspond very rare mineral to а (W)  $NaCa(Mg,Fe,Mn,Al)_5Si_8O_{22}(OH)_2$ , which according to the intensity of main reflection is represented by 45.6%, and is classified like monoclinic amphibole. Beside that this sample has feldspate (F - 23.60%,) quartz (Q - 16.4%), calcite (C - 8.90%) and clay minerals - 5.6 %. Among clay minerals prevails montmorilonite, then mixed layers ilite-montmorilonite and montmorilonite-chlorite. Minimal amounts of mica and ilite are noticed [Figure-13].



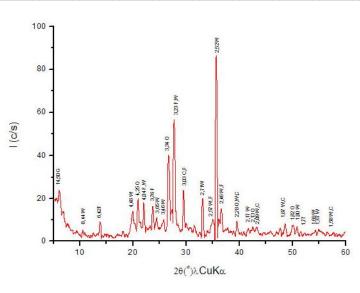


Fig. 13: Profile 8 - C (140-155 cm)

## [IV] CONCLUSION

Results of the laboratory research show that quartz and feldspates are mainly present in sand and silt fractions in mineralogy of researched vertisols. According to the studied vertisols the mineralogy is inherited from the parent material and it does not significantly altered with pedogenesis. Primary and secondary minerals in parent material are created by decomposition of different rocks (mainly basic) that are found above the lake basins. The exception is profile 6 (andesite brecca) where in some horizons a very rare minerals with high percentage can be met (koashvite, winchite), which show high resistance to the chemical decomposition. Unlike the bigger fractions, monmorrilonite and mixed-layer minerals (ilitemonmorrilonite) dominate in the clay fraction and there are smaller quanities of ilite and chlorite.

The preliminary results of this study confirm the further characteristics of these rare minerals, using other methods such as scanning electron microscopy, SEM, ICP-MS and so on. %.

#### FINANCIAL DISCLOSURE

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#### CONFLICT OF INTERESTS

The authors declare no conflict of Interests

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