



## Hardsetting soil in alluvial fan sediments at Sinjar area, northwestern Iraq: mineralogy and geochemistry

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**ABSTRACT** - Hardsetting soil is recorded for the first time in Iraq within a claystone bed about 2 meter thick, which is developed mostly as alluvium deposits. A dry medium tough claystone bed collapses, fragmented, and becomes powder within few seconds upon wetting with hearable structural fragmentation. Preliminary mineralogical, chemical and morphological investigations using X-ray diffraction, X-ray fluorescence and scanning electron microscopy have revealed that the mineralogical composition includes clay minerals as chlorite, kaolinite, and illite and non-clay minerals as quartz, feldspar (albite), and calcite. These mineral phases affect on the distribution of major and trace elements that were associated with or adsorbed to most of them especially clay minerals. The morphological characteristics of the clay fraction indicate their re-transportation and re-deposition from older clastics in the region. The studied soil was formed pedogenically as slurry in erosional lag concentrates weathered from marl and claystone of the Miocene Fatha and Injana formations.

**KEY WORDS:** Hardsetting soil, alluvium sediments, Sinjar area, Iraq

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### INTRODUCTION

The term "Hardsetting" soil is defined by Mullins (2000) as soils that undergo structural breakdown during wetting and then set to a hard structureless mass during drying. This term is now widely used worldwide. In terms of their world wide distribution, the low concentration of soil-texture organic matter (SOM) expected in hardsetting soils means that they are most likely to occur in arid and semi-arid tropical and mediterranean areas (Mullins, 2000).

Hardsetting soil has been related to two main processes: a) development of strength when the soil is still moist due to matric suction acting within interparticle and interaggregate bridges, and b) temporary cementation of dry soil by poorly ordered silica and aluminosilicates (Bresson and Moran, 2005).

Hardsetting soil is difficult or impossible to cultivate until it is rewetted. It has low infiltration and much of the rain runs off. The particle size range and lack of organic matter determine their behavior. For a soil to hardest, two things must happen; firstly, the soil must collapse or slump on wetting and lose any open structure it may have, secondly, as the soil dries, it must not crack and restore an open structure, but instead set into a featureless mass (Daniells, 2009).

Where hardsetting soils exist, their behavior commonly exerts a dominant influence on soil management and crop productivity and may result in serious environmental problems where it results in runoff and erosion (Mullins, 2000).

The most important impacts that a hardsetting has in a soil is its effect on root growth and the water regime of the soil. Hardsetting soils restrict the down growth of roots through restricted root penetration and the creation of seasonal saturated conditions due to the low permeability of the

hardsetting. Water mound can build up above such soil in condition similar to septic tank drainage fields (Daniels and Friton, 1994).

In Iraq, no indications for hardsetting soils occurrences are recorded. In the present study, a claystone bed about 1.8-2.2 m thick appeared in a hand-dug well near Sinoni village, north of Sinjar mountain (Fig. 1), is tough to medium tough when it is dry and when wet the sample collapse suddenly and becomes powder within few seconds, with an ability to hear its structural fragmentation through wetting.

The studied soil has developed in parent materials that (i) move along steps of northern limb of Sinjar anticline as collovium and alluvium from the exposed Miocene clastics in particular Fatha and Injana formations (Lower Fars and Upper Fars formations respectively) or (ii) remained in place after deposition within the syncline as fluvial and fluvio-lacustrine deposit of Fatha and Injana clastics.

The aim of the work is to investigate the mineralogy and chemistry of the hardsetting soil horizon firstly mentioned in alluvium at Sinjar area, northwestern Iraq and to discuss its genesis.

### GEOLOGICAL SETTING

The study area is located within the Foothill Zone of unstable shelf of the Nubio-Arabian platform, north of Sinjar anticline by about 5 km. In Sinjar anticline, the exposed rocks ranges in age from Cretaceous to Neogene. They are mostly carbonate except the restricted clastic outcrop of the Fatha Formation and some sandstone and claystone outcrops of Sinjar Formation about 8 km to the north of northern limb of Sinjar anticline.

The present area is located within the broad undulated northern syncline of Sinjar anticline. The dip angle not

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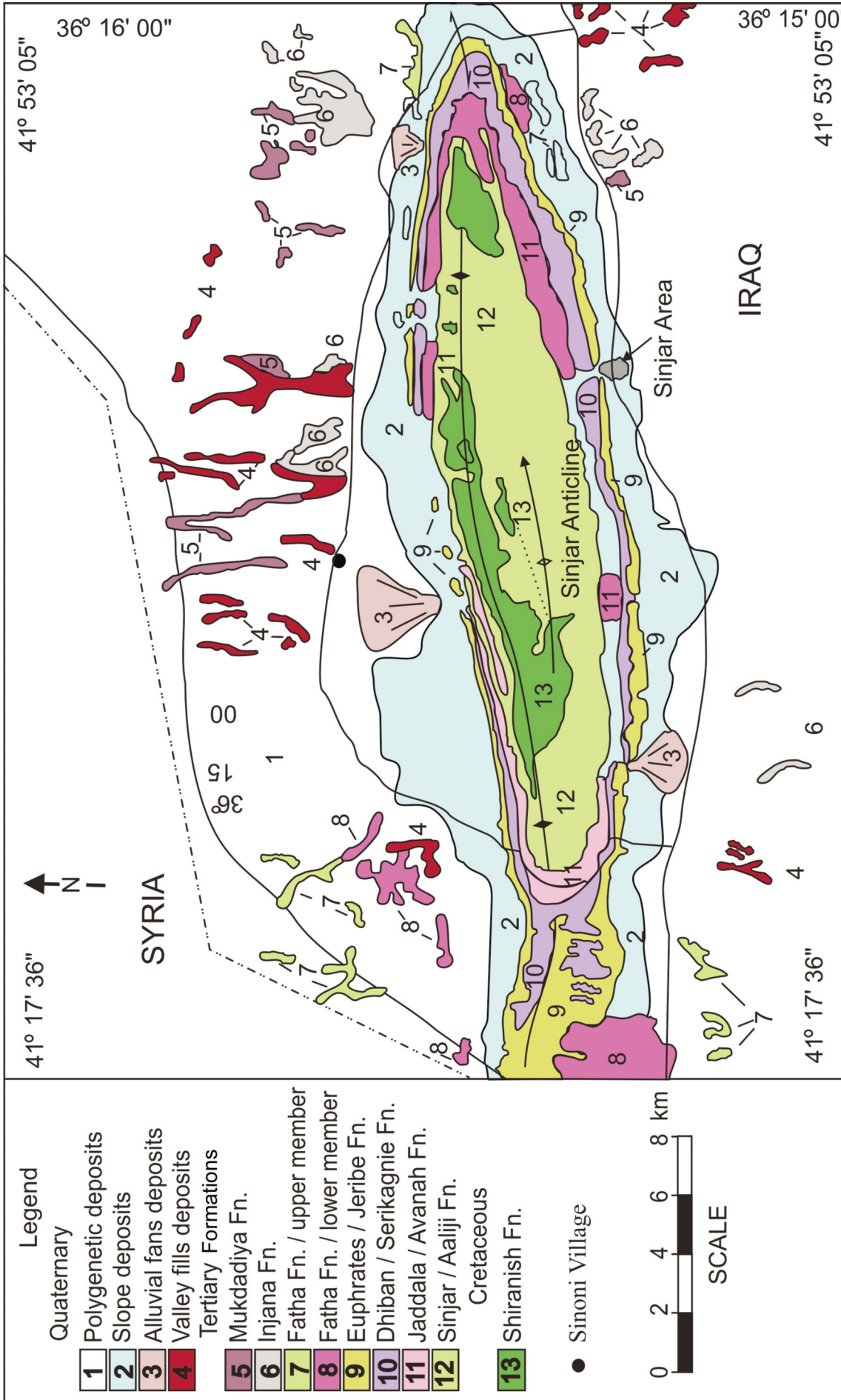


Fig. 1 - Geological map of the study area showing the main geological formations and the main morphological features of the area (modified from Geosurv, 1995).

exceeds 2°N. The most important geomorphological feature in the area is the alluvial fan deposits, which cover most of the northern limb of the anticline (Fig. 1). These fans play an important role in the deposition of the hardsetting soil in question.

Al-Daghastani (1989) divided the fans in Sinjar anticline into active and inactive alluvial fans. Active alluvial fans being the larger. The Gully Karsi fan (in which the study area is located, Fig. 1) was studied in detail by Al-Hamadani et al. (2003) during their ground water investigation of the region. Two types of flow were responsible for deposition: debris flow and mudflow. Debris flow deposits occur in the fan head and mid fan areas, whereas mudflow deposits occur in the proximal and terminal parts of the fan. Distal Gully Karsi fan deposits are fine grained, and generally consists of better sorted stream flow sediments that are porous and permeable.

The greater abundance of mudflow and debris flow deposits in proximal and medial parts of the fan may result in decreased and less predictable porosity and permeability in these areas (Al-Hamadani et al., 2003).

### SAMPLES AND METHODS

Samples were collected as channel samples from a hand-dug well near Sinoni village, north of Sinjar area at a depth of 2.5 meters. They are light brown and relatively tough soil fragments (peds).

X-ray powder diffractogram was made on bulk samples to determine overall mineral composition using Phillips Spellman DF3 diffractometer with Ni-filter Cu- $\alpha$  radiation at 40Kv and 20 mA. Specimens were scanned at 2° 2 $\theta$  to 40° 2 $\theta$ . The bulk composition were determined based on method of Moore and Reynolds (1989). Semi-quantitative determination of these mineral constituents are made using SIROQUANT V3 program. A Siroquant version 3 is a semi quantitative phase

analysis software for windows from X-ray powder diffraction patterns of mixtures. The program is released by the CSIRO (Commonwealth Scientific and Industrial Research Organization), Australia in 2006.

Samples were gold-coated for scanning electron microscope (SEM) analysis. A JEOL JSM 6460A scanning electron microscope is conducted using camscan electron microscope. Furthermore, additional scanning electron microscopy analysis is achieved at the Steinmann Institute for Paleontology of Bonn University, Germany, using CamScan MV2300 for better determination of the studied soil. The chemical analysis for major and trace element of the claystone horizon were achieved using XRF Fluorescence technique and were run on a Spectro Xepos spectrometer. All these analyses were achieved at laboratories of Wollongong University, Australia.

### MINERALOGY

The main clay minerals observed are kaolinite, illite, and chlorite; the non-clay minerals include quartz, feldspars (albite), and calcite (Fig. 2). The result of semi-quantitative X-ray diffraction analysis is shown in Tab.1. The mineral constituents are commonly observed in the Injana and Fatha formations (Al-Juboury, 2001; Al-Juboury and McCann, 2008).

Clay minerals are either of terrigenous (detrital) and/or authigenic and diagenetic in origin. Kaolinite seems to be of detrital origin derived mainly from igneous rocks rich in alkali feldspars, and to lesser extent from the reworking of older sedimentary rocks (Millott, 1970). The presence of kaolinite as a subsidiary mineral is an indication of relatively little leaching effect and chemical weathering in the source area (Chamley, 1989). Hexagonal plates with pitted surfaces and eroded plates (Figs 3C, E and 4) could be interpreted as re-transported and re-deposited (detrital) kaolinite (Murray, 1976; Keller, 1978).

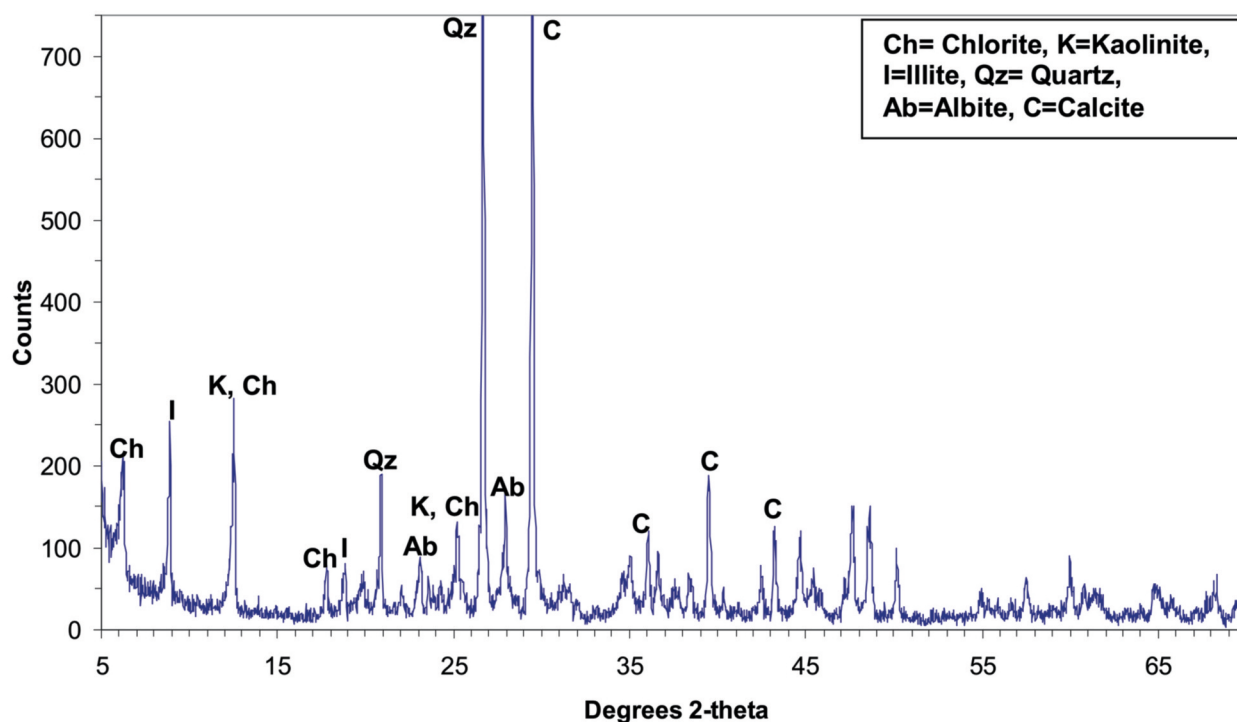


Fig. 2 - X-ray diffractogram illustrating the presence of clay and non-clay mineral phases in bulk rock.

Minerals	XRD Semi - quantitative (%)	Chemical analysis (%)
Calcite	33	30
Quartz	20	11
Albite	9	16
Mg-chlorite	15	12.5
Fe-chlorite		7.5
Illite	17	19
Kaolinite	5	4
Total	100	100

Tab. 1 - Mineral concentration according to semi quantitative XRD analysis and normative calculation of chemical analysis.

Illite could be formed as a result of alteration of muscovite, biotite, and k-feldspar both in weathering zone and during diagenesis (Hower et al., 1963). In the present study, illite occurs as fibers or as crusts (Fig. 3D) which may indicate the altered form of illite from older feldspars or other silicate minerals.

Chlorite is derived from the weathering of rocks rich in ferromagnesian minerals that contains high Mg and Fe and that is excellent in the basic igneous and metamorphic rocks (Millott, 1970).

Scanning electron images show that the studied hardsetting soil are fractured (Fig. 3A) with close-packing arrangement of the particles with usual presence of voids and massive microstructures (Figs 3 and 4) and linear feature that could be root or fungal hyphae traces (Figs 3F and 4b).

Fe-chlorite gives strong second and fourth basal reflections but weak first and third basal reflections, while Mg-chlorite gives strong first basal reflections (Curtis et al., 1984; 1985; Segall et al., 1987; Moore and Reynolds, 1989). According to the pattern of x-ray diffraction it may be looks that there are a mix of Mg-chlorite and Fe-chlorite reflection especially 1<sup>st</sup> and 2<sup>nd</sup> reflections.

Tab. 1 mentioned that the mineral composition of chlorite is about 19% (12% Mg-chlorite and 7% Fe-chlorite). Fe-chlorite associated with sedimentary rocks is preserved through neof ormation (Weaver and Pollard, 1975). The mole fraction of Mg decreases in chlorite as this mineral was transported from a fluvial environment into a normal marine environment (Segall et al., 1987). The chemical formula of Mg rich chlorite [(Mg, Al)<sub>10</sub> Fe<sub>2</sub>(Si, Al)<sub>8</sub> O<sub>20</sub>(OH)<sub>16</sub>] could be changed to Fe rich chlorite [(Mg, Al)<sub>6</sub> Fe<sub>6</sub>(Si, Al)<sub>8</sub> O<sub>20</sub>(OH)<sub>16</sub>] (Brindly and Brown, 1980). Fe-chlorite among many clay minerals forms by aggradation of soluble cations in alkaline confined environment (e.g. lagoon). The neof ormation of Fe-chlorite takes place by the addition of iron and a little aluminum to degraded clay types (Millott, 1970). This happens commonly under reducing condition where Fe becomes mobile.

## CHEMISTRY

The chemical analysis in Tab. 2 shows that the most MgO is due to the clay minerals as Mg-chlorite, whereas, CaO belongs to calcite (Fig. 2).

Al<sub>2</sub>O<sub>3</sub> distributed among albite-plagioclase, illite and kaolinite. Albite represents the parent aluminum-silicate minerals where clay minerals derived from. Kaolinite may occur as an inherited mineral from the source area (Fig. 3A).

Na<sub>2</sub>O related to albite-plagioclase, while the concentration of K<sub>2</sub>O exceeds 2% reflected illite; it may be reconstituted by K-fixation of degraded illite due to high potassium activity of concentrated sea water (Fisher, 1988). Illite and (chlorite) are the common detrital minerals inherited by numerous soils and sediments, and they may be stable during weathering with minimum physical and chemical activities free of cations (Millott, 1970).

Silica distributed between igneous and clay minerals as main components in addition to quartz. Quartz transported as very fine particles associated with the clay grains through the weathering and leaching processes to various depositional environments.

From the chemical analysis the residual amount of Fe may relate to iron oxide-hydroxides phases (e.g. limonite-goethite) which reflected the yellow to yellowish grey color of the marl beds. MnO is associated with the clay minerals as product of weathering and diagenetic processes, as well as Mn substituted in Fe-oxy-hydroxides phases.

TiO<sub>2</sub> found in many suites in igneous and clay minerals substituted in Si or Al suites and /or as districted minerals like anatase and rutile.

Iron oxides-hydroxides (hematite and goethite), as well as other minerals illite, Ti-oxides phases (usually anatase and rutile) and quartz are associated due to the their similar geochemical behavior through weathering, leaching intensity, transportation and sedimentation processes on the parent igneous and metamorphic rocks. The differences in their quantities may be due to the diagenetic process, which redistribute mineral phases in their sedimentary environments under different climatic conditions. (Ibanga et al., 1983).

Trace elements are distributed in marl deposits in many phases: 1) substituted the major elements e.g V, Cr and Y in Al site in igneous and clay minerals, Rb in K site in illite, Sr, Ba, Y and Zn in Ca site in carbonate minerals like calcite; 2) adsorbed on clay minerals like: Ni, Cu and Zn, or iron oxides like: Ni, Cu and Co; 3) as districted mineral e.g. Zr in Zircon; 4) may be found as very little amount of secondary minerals like S in secondary gypsum and Cl in halite.

## DISCUSSION

The present hardsetting soil may be deposited as heavy and thick liquid slurry in erosional lag concentrates weathered from marl and claystone of the Fatha and Injana formations. Physical ripening of these weathered materials is due to close packing of their silty and fine-sand particles as observed by SEM micro images. The common presence of fine sand and silt particles is not surprising given the tendency for the studied hardsetting soil to be particularly structurally unstable similar to some studied soils worldwide (like Australian red brown earth; Cockroft and Martin, 1981; Franzmeier et al., 1996). Clay minerals composition (dominance of hydrous mica and/or kaolinite) may be responsible for instability of hardsetting with a smaller shrink/swell potential (Norrish and Pickering, 1983) similar to the present soil in which kaolinite and illite are common constituents of their clay mineral composition.

Desiccation or fracturing in the present hardsetting soil (Figs 3A and 4A-B) and close-packing nature with retaining

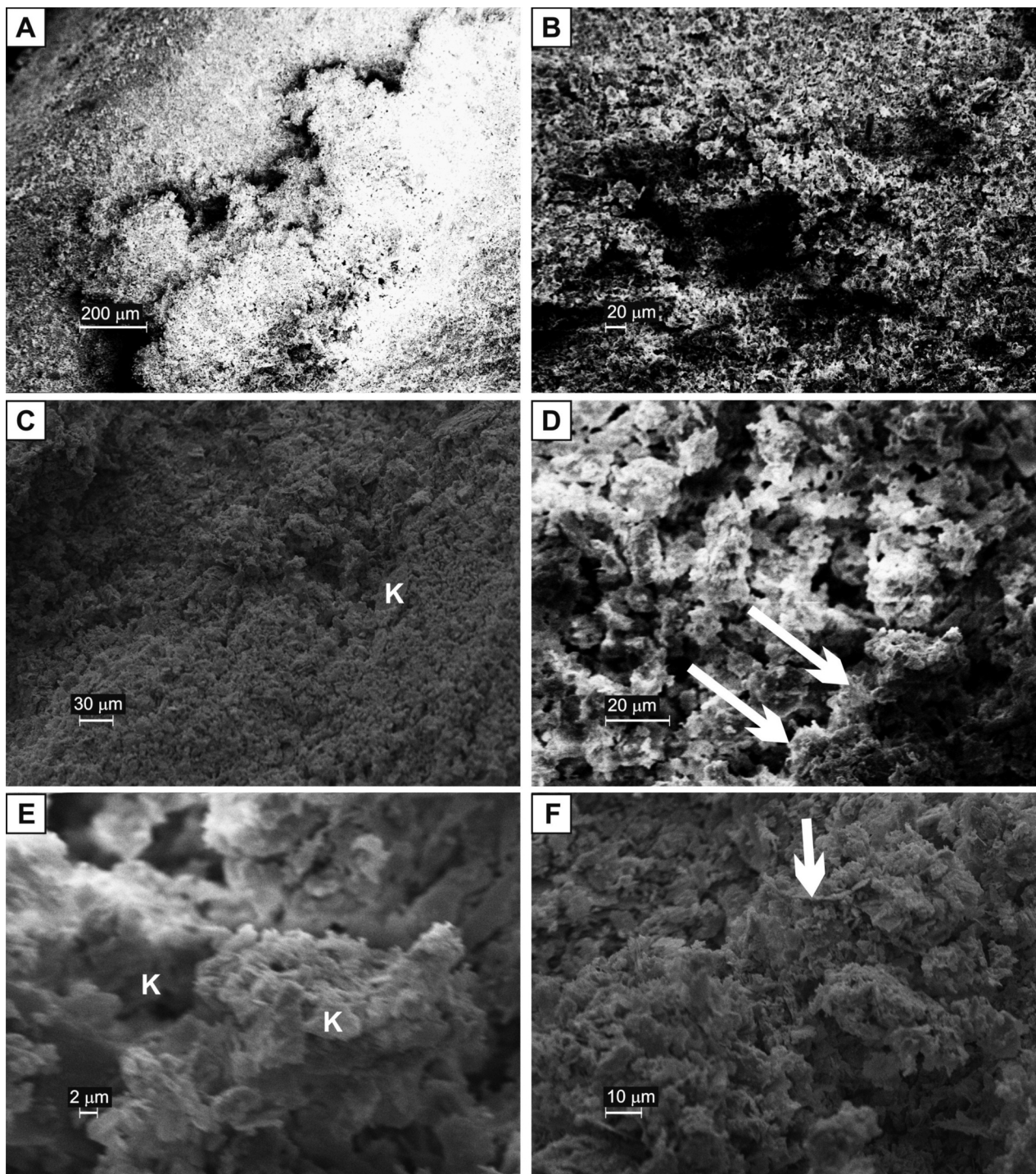


Fig. 3 - Scanning electron microscopic images illustrating select micromorphological features of the studied hardsetting soil. A- Surfaced void and fractures coat silt and clay-sized particles. B- Close-packing distribution pattern with scattered void. C- Closely packed fabrics (silt grains are closely packed with relatively clean very fine sand, silt, and clay-sized particles (or clay minerals, degraded or erode plate of kaolinite (k) is observed, voids are primarily packing voids. D- Irregular surface with discontinuous voids, note fibrous illite ( arrows). E-F- Dense massive and closely packing particles with scattered eroded kaolinite plates (k), linear feature partly curved (arrow) may be root or fungal hyphae traces.

of initial clay-bridging as illite fibers (Fig. 3D), a close-packing arrangement of the particles (mostly of fine sand and silt) of the present soil (Fig. 3) could be responsible for its hardness. It may be also due to presence of inorganic cements (e.g. clay minerals). Fragmentation by wetting or humidity is promoted also through several voids and fractures (Fig. 3A-B). The periodicity and rapidity of profile wetting and drying may have influenced structural development in the studied soil

horizon as mentioned in other reviews (Chadwick and Graham, 2000).

**CONCLUSIONS**

Mineralogical and petrological (X-ray diffraction and scanning electron microscopy) and bulk-rock geochemistry (X-ray fluorescence) of hardsetting soil is studied for the first

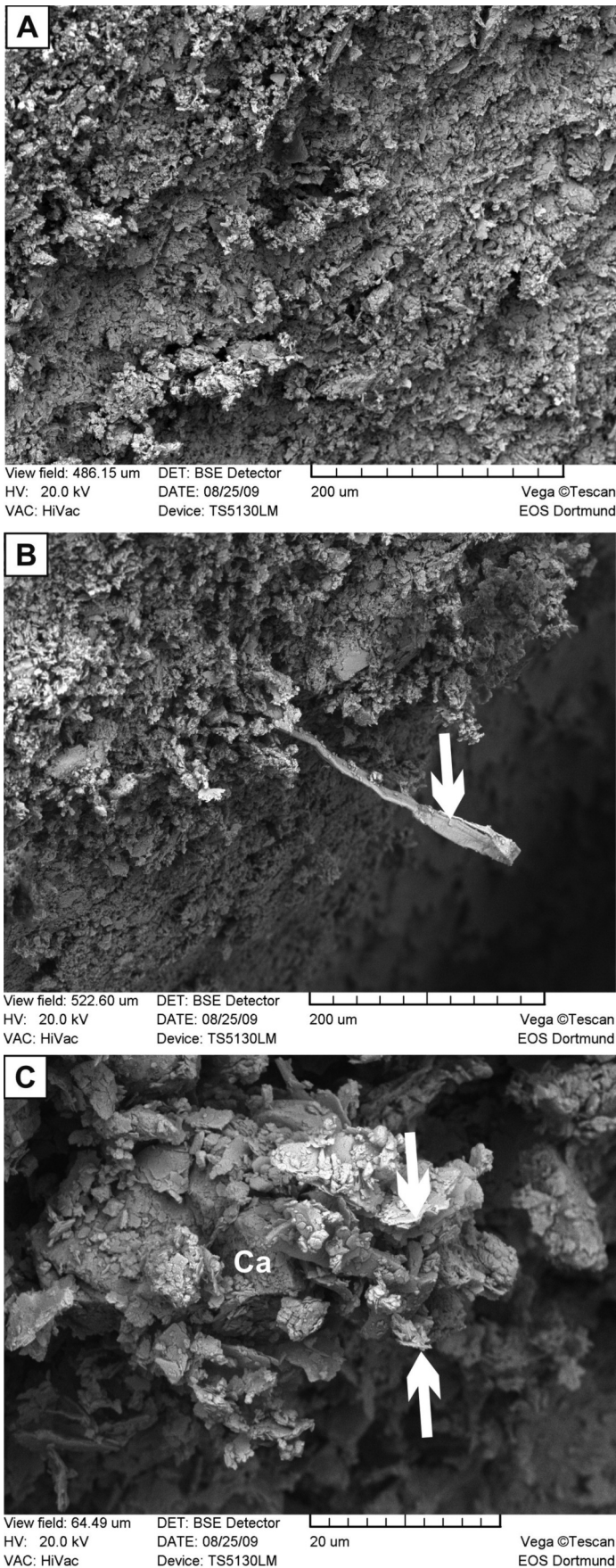


Fig. 4 - A- General view of the hardsetting soil showing the directional trend of its compositional constituents with common fracturing, pores and cavities. B- Common microstructures and voids with few root? (arrow) penetrating soil. C- Carbonates (Ca) and clay minerals (kaolinite, arrows) in hardsetting soil studied.

Oxides	Wt%	Trace elements	ppm
SiO <sub>2</sub>	38.98	S	320
TiO <sub>2</sub>	0.62	Cl	2864
Al <sub>2</sub> O <sub>3</sub>	12.85	V	121
Fe <sub>2</sub> O <sub>3</sub>	5.54	Cr	198
MgO	5.36	Ni	135
CaO	16.91	Co	19
Na <sub>2</sub> O	1.88	Cu	36
K <sub>2</sub> O	2.11	Rb	75
MnO	0.11	Zr	110
L.O.I	15.49	Zn	81
Total	100.00	Sr	235
		Ba	347
		Y	18
		Pb	9

Tab. 2 - The chemical analysis (major oxides and trace elements).

time in Iraq. These analyses have revealed that such soils are mostly fractured and associated with fine sand and silty particles due to their deposition as heavy and thick liquids (slurry) in erosional lag concentrates. Their clay mineralogy is dominated by kaolinite and illite which are suggested to be a cause for their instability.

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