



## REEVALUATION OF KAOLINITE OCCURRENCE IN SOME TORRIFLUENTS IRAQI SOIL

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### Abstract

Clay fraction of Torrifluents Iraqi soils contained different clay mineral groups. Usually, samples with limited number of clay minerals, can be defined mainly by x-ray diffraction analysis. Whereas poly-mineralized soil needs additional techniques to clarify its mineral composition accurately. Kaolinite which is characterized by reflection peaks at  $7.2 \text{ \AA}^\circ$  and  $(3.55\text{-}3.57 \text{ \AA}^\circ)$ , are normally overlapped with those of chlorite peaks. This work was carried out to reevaluate the occurrence of kaolinite in some Iraqi soils with the presence of chlorites. Clay fractions from four Torrifluents Iraqi soils were used in this study. X-ray diffraction analysis with different heat treatments is referred to the presence of kaolinite and soil chlorite. Intercalation with dimethylsulfoxide, after saturation with Li (Li-DMSO) showed the presence of a peak at  $11.3 \text{ \AA}^\circ$ , which confirmed the presence of trace amount of kaolinite. However, the continuous occurrence of the peaks at  $7.2$  and  $14.4 \text{ \AA}^\circ$  are referred to the presence of chlorites. Reduction the intensity of  $7.2 \text{ \AA}^\circ$  peaks by heating at temperature below  $500^\circ \text{C}$  confirmed the presence of soil chlorite which overlap with kaolinite. There was no difference among the four soils with regard to the presence of soil chlorite and kaolinite.

**Keywords:** Kaolinite, Chlorite, Torrifluents soil, Iraq.

## إعادة تقييم وجود الكاؤولينيت في بعض الترب الرسوبية العراقية

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### المخلص

يحتوي مفصول الطين في الترب الرسوبية العراقية على عدد من معادن الطين، وعادة يمكن تحديد التكوين المعدني للعينات التي تحتوي على عدد محدود من معادن الطين بشكل رئيس باستخدام حيود الأشعة السينية، بينما العينات التي تحتوي على معادن متعددة تحتاج الى استخدام تقانات اضافية للتعرف على تكوينها المعدني بدقة. يتميز الكاؤولينيت بقمم حيود الاشعة السينية عند 7.2 أنكستروم و (3.55-3.57) أنكستروم والتي عادة تتداخل مع تلك الخاصة بالكوريت. أجري هذا العمل لإعادة تقييم وجود الكاؤولينيت مع وجود الكوريت في بعض الترب الرسوبية العراقية. استخدم في هذه الدراسة الجزء الطيني لأربعة ترب رسوبية. أظهرت نتائج تحليل حيود الأشعة السينية مع استخدام عدة معاملات حرارية مختلفة وجود الكاؤولينيت مع كلوريت التربة. أظهرت نتائج التشبيح بالليثيوم والمعاملة بثنائي مثل سلفواوكسايد وجود قمة انعكاس عند 11.3 أنكستروم والتي تؤكد وجود كمية ضئيلة من الكاؤولينيت. استمرار وجود الانعكاس عند 14.4 و 7.2 أنكستروم يوضح وجود الكوريت. انخفاض شدة الانعكاس عند 7.2 أنكستروم عند التسخين لدرجات حرارة أقل من 500 °م يؤكد وجود كلوريت التربة الذي يتداخل مع الكاؤولينيت عند التشخيص. لم يظهر أي فرق بين عينات الدراسة الأربع فيما يخص وجود كلوريت التربة والكاؤولينيت.

كلمات مفتاحية: كاؤولينيت، كلوريت، ترب رسوبية، العراق.

### Introduction

Studies of soils, showed that clay fraction of Torrifluvents Iraqi soils contained a mixture of several clay minerals i.e. smectite, chlorite, mica, vermiculite, kaolinite and palygorskite; in addition to some regular and irregular stratified layers (1, 3, 10). Most studies reported the dominancy of smectite, followed by mica or chlorite (2, 12), whereas others have mentioned that chlorite or chloritized-montmorillonite or chloritized-vermiculite was the dominant clay mineral (4, 8).

There was a contradiction between these studies in the ratio of kaolinite in these soils type. Some of these studies indicated that it is the third component, while others reported that its ratio is low and it is between 5-10% in Iraqi alluvial soils. This contradiction is probably due to the difficulties to recognize the mixture of clay minerals components in the same sample using routine x-ray diffraction analysis (XRDA). The presence of several clay minerals do not give discrete diffraction peak

and/or some peaks are over-lapping. This is the case with kaolinite and different types of chlorite, when they are presented in the same sample. Various chemical and physical techniques can be applied prior to XRDA to overcome differentiation problems among these two minerals. Routine differentiation among kaolinite and chlorite minerals using XRD usually depends on the resistance or disappearance of 7.2 and (3.55-3.57) Å peaks after specimen heating to 500 °C for 2 hrs. Other techniques to distinguish among kaolinite and chlorite have used boiling with NaOH or treatment with hot HCl or intercalation with KOAC, hydrazine or Li-DMSO (6, 9).

This work was aimed to reevaluate kaolinite occurrence and differentiate among kaolinite and chlorite in the clay fraction of some Torrifluents Iraqi soils by using some of these mentioned techniques with XRD.

### Materials and Methods

Surface soil samples from four Typic Torrifluents Iraqi soils and kaolinite sample from Iraqi kaolinite deposit mineral are used in this study. These soils formed from Euphrates alluvium (S1 and S2) and Tigris alluvium (S3 and S4). Clay dispersion is carried out after cementing agent removal. Carbonate minerals are removed by 1N NaOAC at pH 5, organic matter is destroyed by oxidation with NaOCl, pH 9.5, whereas, free iron and manganese oxides are removed using CBD (sodium citrate-bicarbonate-dithionite) method (7). Clay fractions are separated by centrifugation. Clay samples are saturated with Mg<sup>2+</sup> and K<sup>+</sup> using 1 N of MgCl<sub>2</sub> and 1N of KCl, then washed with distilled water and ethyl alcohol until it becomes free of chloride. Specimen of air-dry Mg-saturated and K-saturated are examined using x-ray diffractometer. K-saturated specimen also examined after heating to 110, 300, 400, 500 and 550 °C for 2 hrs. Non-dried parts of these samples are also solvated with Dimethylsulfoxide (DMSO) after lithium saturation using the modified procedure of Lim *et al.*, (9). Samples also treated with 2 M HCl at 80 °C for 24 hrs. to dissolve chlorite clay mineral (11) and then, examined by X-ray. X-ray patterns are obtained by using Phillips PW 1050 x-ray diffractometer working at 40 kV and 20 mA. Nickel filtered copper- $\alpha$  radiation was used with a goniometer speed 2° (2 $\theta$ ) per 2 minutes.

### Results and Discussion

The X-ray diffraction traces revealed that the studied clay fractions are poly-mineralised. X-ray diffraction patterns of Mg-air dried showed a peak at 14.4 Å. This peak may be contributed to smectite, chlorite and vermiculite. Mg-saturated specimen and solvated with ethylene glycol showed swelling to 17.0 Å spacing (data not shown here). This peak is contributed to smectite mineral. The 10 Å basal spacing at Mg-air dry and K-air dry and its persisting with heating treatment confirmed the presence of illite clay mineral (Fig. 1).

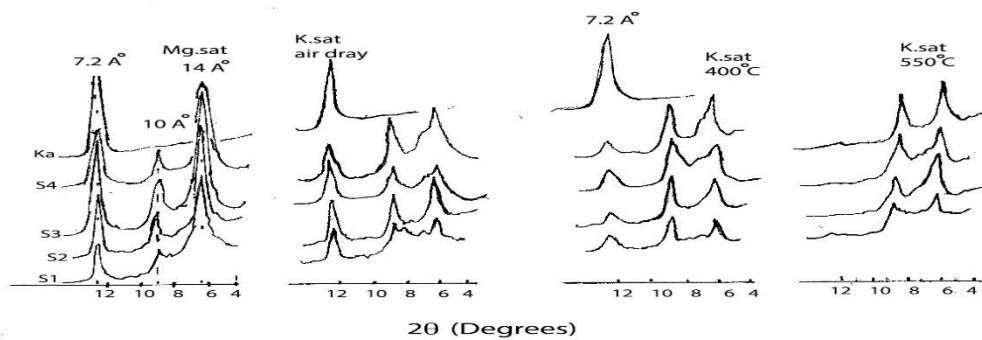


Fig. 1. X-ray diffraction traces of Mg-saturated, K-saturated and K-saturated and heated for kaolinite (Ka) and clay fraction of the studied soil

The presence of 7.2 Å peaks at x-ray diffraction patterns of Mg-air dry, Mg saturated and ethylene glycol solvated, K-air dry and K-550 °C heating are related to both kaolinite and chlorite minerals. The presence of these two minerals together led to a problem how to distinguish among them. Layers of highly charged clay minerals (smectite and vermiculite) collapse after potassium saturation and heating to 110 °C. Thus, definable 14.4 Å peak at this treatment confirmed the presence of chlorites.

However, kaolinite should be destroyed by heating above than 500 °C, which cause complete disappearance of its first order peak at 7.2 Å. X-ray diffraction traces of the studied samples showed clear differences in this peak intensity after heating to 300 °C and above (data for heating to 300, 450, and 500 °C not shown here). This decrement in 7.2 Å intensity may be related to the presence of non-heat resistant type of chlorite (soil chlorite).

The presence of kaolinite and non-heat resistance chlorite in soil led to a problem in kaolinite identification because 7.2 Å peak which is given by both minerals are affected by heating. So, it is necessary to use other technique(s) to overcome this problem. Intercalating procedures with different chemical materials was a successful one. The treatment with Li-DMSO resulted in a complete expansion of kaolinite layer. The mechanism of DMSO in opening the layers of kaolinite is by breaking the internal bonds and forming a hydrogen bonds with all oxygen atoms and hydroxyl group of tetrahedral and octahedral layers, respectively (6, 9). The results showed a complete expansion of the 7.2 Å x-ray diffraction to 11.3 Å (Fig. 2). The low intensity of 11.3 Å peaks in clay samples of the studied soils clearly reflected the presence of trace amount of kaolinite in these soils. However, a large x-ray diffraction peak at 7.2 Å and a peak at 14.4 Å for all examined soil clay still remained after treatment with Li-DMSO. This confirmed the presence of chlorite.

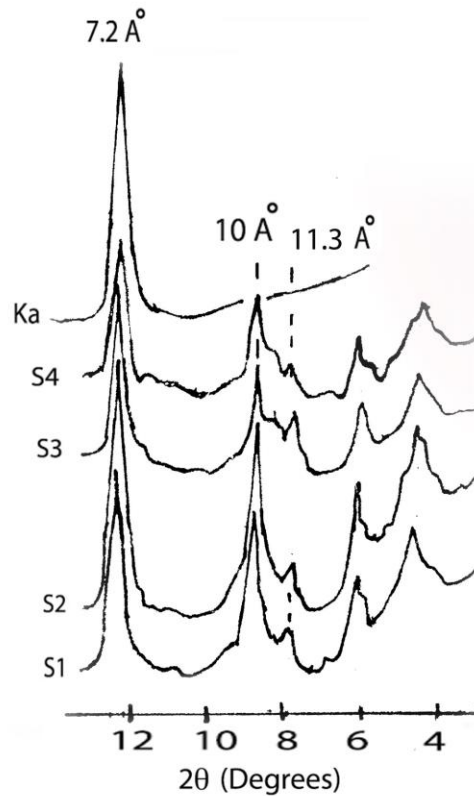


Fig. 2. X-ray diffraction traces explained the complete expansion of kaolinite after Li-DMSO treatment

Stability of clay minerals in the studied samples after treatment with hot HCl is illustrated in Fig 3. Results indicated the disappearance of the  $14.4 \text{ \AA}$  and with the high reduction in the  $7.2 \text{ \AA}$  basal spacing. That is due to the removal of chlorite. Also, a noticeable reduction in  $7.2 \text{ \AA}$  peak intensity is found at heating less than  $550 \text{ }^\circ\text{C}$ .

Heating of K-saturated specimens caused a reduction in  $7.2 \text{ \AA}$  peak intensity, while the intensity of  $14.4 \text{ \AA}$  peak remained unchanged (Fig. 1). These results confirmed the presence of soil chlorite, which may be Fe-rich or maybe swelling chlorite (5). On the other hand, the  $14.4 \text{ \AA}$  diffraction peak persists after heating at  $550 \text{ }^\circ\text{C}$  confirming the presence of chlorite. Thus, the presence of kaolinite with the true chlorite and soil chlorite in the studied samples make it more difficult to differentiate among by heating treatments.

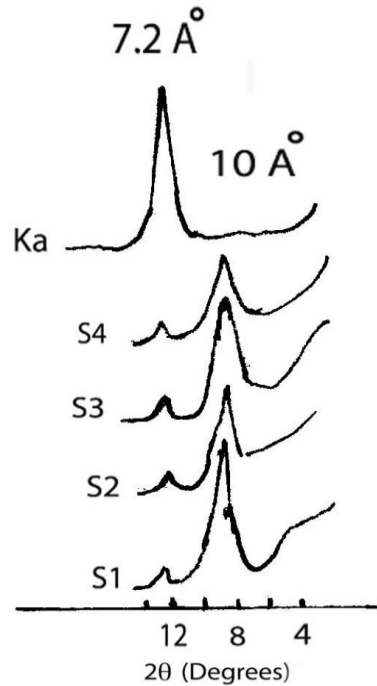


Fig. 3. X-ray diffraction traces after treatment with hot 2 M HCl

In general, one can conclude that it cannot rely upon heating treatment to 550 °C only to distinguish among kaolinite and chlorite and to calculate kaolinite percent in the clay fraction of Torrifluvents Iraqi soil. Thus, using Li-DMSO intercalation or other techniques is necessary to identify kaolinite in these soils. There was no difference among the studied soils which formed from Tigris and Euphrates alluvium material with regard to the presence of soil chlorite and kaolinite.

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