

**Qualitative and Semi Quantitative Analysis of Sample from Kolosh Formation
in Kurdistan Region North of Iraq**

Tahir Ahmad Tahir and Omer Fikrat Bahjet

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Formation in Kurdistan Region North of Iraq**

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Abstract

A sample from Kolosh formation nearby Hizop small village near Kaniwatman area in Kurdistan region was taken for the purpose of conducting qualitative and semi-quantitative analysis for most of elements that constitute the sample. Initial simple physical examination shows that the sample contains a percentage of iron (III) oxide. Three parts of the sample were allowed to react with three in excess concentrated hydrochloric, nitric and sulphuric acids separately. The reaction with HCl gives deep yellow solution, with HNO₃ and with H₂SO₄ gives colorless solution. Most of the transition elements in the periodic table were excluded for the absences of the characteristic colors of their ions in solutions. Analysis shows that the percent of total organic carbon (TOC) in the sample was 5.3%, for silica was 26.9% and for iron was 2.5%. The percentages of missing weight from the samples after their reaction with sulphuric, nitric and hydrochloric, acids were, 42.3%, 44.3% and 48.7% respectively. The group's qualitative analysis for Kolosh sample showed a measureable presence of iron, silicon, carbon and calcium compounds. The presence was very low for other elements compound like barium, aluminum, lead and thallium. The aim of this work is a qualitative and semi quantitative analysis for probable existence of some elements compound in Kolosh formation mineral.

Key Words: Kolosh formation, Shale mineral, Calcium carbonate, Calcium sulphate, Silica, Total organic carbon (TOC).

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تحليل وصفي و شبه كمي لنموذج من تكوين كولوش الجيولوجي في اقليم كردستان شمال العراق

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الخلاصة

تم اخذ عينه من تكوين كولوش الواقع في منطقة هيزوب الصغرى القريه من منطقة كانيوتبان (كاني وتبان) في اقليم كردستان لغرض اجراء تحليل نوعي و شبه كمي لاغلب العناصر المكونه للعينه. تبين من الفحص الفيزيائي الاولي ان الخام يحتوي على نسبه من اوكسيد الحديد (III) . تمت مفاعلة ثلاث اجزاء من العينه مع ثلاث حوامض مركزه : الهيدروكلوريك و النتريك و الكبريتيك كل على حده. تم الحصول على محلول اصفرغامق من تفاعل حامض الهيدروكلوريك، و محلول عديم اللون من تفاعل حامض النتريك ، او حامض الكبريتيك. تم استبعاد اغلب العناصر الانتقاليه في الجدول الدوري لعدم ظهور الالوان الخاصه بأيوناتها في المحاليل. بيّن التحليل ان نسبة مجموع الكربون العضوي في العينه تساوي 5.3% ونسبة السليكون تساوي 26.9% ونسبة الحديد تساوي 2.5% و نسبة الاوزان المفقوده من العينات بعد تفاعلها مع حوامض الكبريتيك و النتريك ، و الهيدروكلوريك هي: 42.3% و 46.2% و 48.7% على التوالي . بيّن تحليل الطوائف النوعي للعينه وجود مركبات عناصر الحديد والكربون و الباريوم و الالمنيوم و الكالسيوم و السليكون والرصاص. الهدف من هذا البحث هو الدراسه النوعيه وشبه الكميّه للعناصر المحتمل وجودها في تكوين كولوش الجيولوجي

الكلمات المفتاحيه : تكوين كولوش ، خام شيل ، كربونات الكالسيوم ، كبريتات الكالسيوم ، سليكا ، الكربون العضوي الكلي

Introduction

Kolosh formation was firstly described by Dunnington ^[1] in Kolosh area northern Koisanjaq city, northern Iraq. It consists of black shale's , fine sandstones, fragments of various grain size of green rock, other minerals, especially [quartz](#) and [calcite](#). The ratio of clay to other minerals is variable.^[2] Black shale is one of meager constituent of Kolosh formation ^[3]. According to literature survey, different definitions for black shale were mentioned. ^[2,4,5,6] It is a dark-colored mudrock containing organic matter, silt and clay-size mineral grains that accumulated together. Vine ^[7] pointed out the compositional variations included in this

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definition and the ways that these variations can be used to study black shale. Most shale that immediately meets this color criterion contains 1% or more organic carbon; 2-10% is a common range. Few shale contain more than 20% organic carbon. Pyrolysis yields variable amounts of liquid and gaseous hydrocarbons, the amount depending in part on the nature of the original organic material and in part on subsequent burial history. Black shale units may have beds enriched in metals by factors greater than 50 for Ag element, for example, and greater than 10 for Mo ^[8]. Such increased concentrations of Ag, Mo, Zn, Ni, Cu, Cr, V, and less commonly Co, Se, and U elements are apparent features of only some black shale.^[9] The enriched values are of controversial origin, having been alternatively attributed to input from [hydrothermal](#) fluids during or after sedimentation or to slow accumulation from [sea water](#) over long periods of sedimentation.^[10] Typical levels for Fe in sedimentary rocks are given by Williamson ^[10] as: limestone 0.33%; sandstone 0.98%, shale 4.7%, and banded iron formation 28%. Almost all Iron (III) oxides Fe₂O₃ are ferromagnetic, which means they all attract to magnets. The organic material that gives black shale its distinctive characteristics is derived from living things. Since black shale has accumulated throughout the entire evolution of life on the Earth, the composition of the organic matter in black shale should reflect evolutionary development of living things at the time the shale was deposited.

In soils and sediments, there are three basic forms of carbon that may be present. They are: (1) elemental C, (2) inorganic C, and (3) organic C. The quality of organic matter in sediments is critical to the partitioning and bioavailability of sediment-associated contaminants. For example, Talley et al., 2002 ^[11], demonstrated that although the majority of PAHs (Poly-Aromatic Hydrocarbon) in a dredged sediment were found preferentially on coal-derived particles, the PAHs on the clay/silt sediment fraction were more mobile and available, and thus potentially of greater concern.

Inorganic carbon forms are derived from geologic or soil parent material sources. Inorganic carbon forms are present in soils and sediments typically as carbonates. The two most common carbonate minerals found in soils and sediments are calcite (CaCO₃) and dolomite [CaMg(CO₃)₂] although other forms may be present (e.g., siderite, FeCO₃) depending on where the soils were formed or where the sediment source was located. It should be noted that

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calcite and to some extent, dolomite, may also be present in soils and sediments due to agricultural input (i.e., liming practices). Organic carbon forms are derived from the decomposition of plants and animals. In soils and sediments, a wide variety of organic carbon forms are present and range from freshly deposited litter (e.g., leaves, twigs, branches) to highly decomposed forms such as humus. In addition to the naturally-occurring organic carbon sources are sources that are derived as a result of contamination through anthropogenic activities. The spills or releases of contaminants into the environment increase the total carbon content present in the soil or sediment. Heavy metals are one of the most poisonous and serious groups of pollutants due to their high toxicity, abundance, and ease of accumulation from various plants and animals. It has been accepted that heavy metals can exist in the environment deriving from a variety of natural and anthropogenic sources. The phenomena of erosion, acidification, and weathering processes have brought input of these metals into the environment in a natural way. According to Idris ^[12], the natural occurrence of heavy metals in aquatic environments and their movement through the hydrocycle in addition to the inputs from anthropogenic activities reflect their ubiquity and complexity. Meanwhile, human activities also contribute to the existence of these metals such as industrial processes, agricultural and aquaculture activities, domestic wastes, and emission from vehicles ^[13]. The present study is for qualitative and quantitative analysis of some possible elements present in geological Kolosh formation mineral.

Materials and Equipment

- a- The Kolosh formation sample was collected from Hizop small village near Kaniwatban area, north Suliamani city, Iraq
- b- All reagents and solvents used were available commercially and were used without further purification unless indicated otherwise.
- c- All the glassware were washed with 0.1 M HCl (Washing solution) and properly rinsed with distilled water and dried before use.
- d- Spectrophotometer model T80 – pg instrument, wavelength 200 – 1000 nm were used for absorbance reading at $\lambda_{\max} = 460$ nm.

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Procedure

A- Simple Physical Test

Strong magnet bar was introduced to Kolosh mineral debris before it crushed to fine powder. Many particles of dark brown color were stick to the bar, which indicate the presence of Fe₂O₃ particles within the mineral.

The same experiment was proceeded using regular iron bar but no particles attracted to it, which confirmed the presence of Fe₂O₃ as it has ferromagnetic properties.

B- Samples of Kolosh rocks were crashed to fine powder in porcelain pestle and mortar with unglazed grinding surfaces, and then sifted through nylon mesh 3microne size before further test.

C- Reaction with Acids:

Three samples of Kolosh mineral each of three grams (3gm) were allowed to react with excess of concentrated sulphuric, nitric and hydrochloric acid separately for overnight to assure complete reaction between the acids and Kolosh samples. The mixtures were filtered, and the precipitate washed several times to remove any trace of the acids, then dried at 105 °C for six hrs., cooled in a dissector and weight as shown in table (1). The filtrates were kept for further test.

D – Determination of Silica

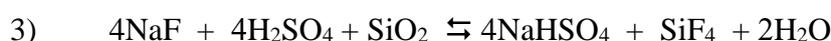
A sample of one gram of sulphurated Kolosh mineral was thoroughly mixed with different weights of sodium fluoride NaF as shown in table (2).

Silica Calculation

Silica present in sulphureted Kolosh were calculated through the following chemical equations:



----- Add



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Referring to figure (1), the plot shows that 1.3 g of NaF was enough to equate and react with all silica present in the sample.

According to equation (3), four moles of NaF react with one mole of SiO₂.

Molecular weight of NaF = 42g/mole, for SiO₂ = 60.1g/mole,

Therefore 1.3g of NaF = $(1.3/42) = 0.031$ moles

Number of SiO₂ moles in sulphureted Kolosh = $(0.031 \times 4) = 7.75 \times 10^{-3}$

$7.75 \times 10^{-3} \times 60.1 = 0.466$ g of sulphureted Kolosh

Kolosh sample lost 27% of its original weight after its reaction with H₂SO₄ (conc.),

Therefore: $(0.466 \times 100) / 27 = 1.73$ g original weight of the sample

Assuming that SiO₂ was not affected by initial reaction of the sample with H₂SO₄ (conc.), the percentage of SiO₂ in Kolosh mineral:

$$(0.466 \times 100) / 1.73 = 26.9\%$$

Percentage of reacted material at the initial reaction of the sample with H₂SO₄ (conc.) = 42.3%

Percentage of reacted silica in the sample with HF = 26.9%

Percentage of the remaining material, = 9.8% which is mostly sulphates that not reacted with hydrofluoric acid

E – Determination of Total Organic Carbon (TOC)

Kolosh sample includes inorganic sources of carbon such as carbonates and most notably calcite, dolomite, and siderite. These minerals react with acids, generating carbon dioxide and corresponding salt. Thus, the determination of total organic carbon (TOC) in Kolosh sample starts by reacting almost 20g of the sample with excess concentrated nitric acid, then concentrated hydrochloric acid to change all inorganic carbon component of the sample to soluble nitrates or chlorides which were discarded later by filtration. The precipitate was thoroughly washed with distilled water and dried at 120 °C for six hours, then cooled in desiccator for overnight. Precisely seven grams of the dry, cooled precipitate was heated for six hours in crucible by naked flames of two torches, one beneath, the other was above the crucible so that the naked flames easily touched all parts of the precipitate in order to burn and maintain the oxidation of any organic carbon particles to carbon dioxide. The color of the

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precipitate gradually turned from dark olive green (fig.2-A) to yellow-creamy color (fig.2-B) as shown in figure (2). The precipitate was cooled in desiccator for overnight, and then its weight was recorded.

This process was repeated three times for same precipitate to ensure that more heating would not cause further loss in the weight of the precipitate. The percentage of TOC in the sample was (5.3%).

F – Determination of Iron

Two grams of iron wool was dissolved in excess of concentrated nitric acid. No residue or precipitate was left after dilution and filtration of the reactants. The filtrate was diluted to 250 ml in volumetric flask. Six serial volumes of this solution were diluted to 50 ml. in volumetric flask as showed in table 3 Solution containing 250 mg/mL of potassium thiocyanate was prepared. One mL of this solution was added to aforementioned six different concentrations. The Fe^{3+} ions react with SCN^- ion to form a blood-red coloured complex as shown in the following chemical equation:



As near as possible to 15 minutes after adding thiocyanate, the absorbance at wavelength of 460 nm for each coloured solution was measured using colorimeter. The absorbance of each six different concentrations of the solutions were measured, plot of Abs. versus complex concentration was drawn as shown in figure (3). The line of best fit for the data points goes through the origin, because absorbance must be zero when Fe^{3+} concentration is zero. The plot confirms that the dilute deep blood red colored solution of complex $[\text{Fe}(\text{NCS})(\text{H}_2\text{O})_5]^{2+}$ obeys Beer's law^[14]

For determination of iron concentration in Kolosh mineral, 5 g sample of powdered Kolosh mineral was allowed to react with excess concentrated nitric acid and left over night for reaction completion. The reactants mixture was filtrated and the filtrate was diluted to 250mL in volumetric flask (Solution A). One mL of Kolosh solution (A) and one mL of potassium

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thiocyanate solution was diluted to fifty mL in volumetric flask (Solution B). The absorbance of new colored solution was measured at $\lambda = 460\text{nm}$.

Iron Calculation

According to straight line equation $Y = mx + b$

Our spread sheet gives: $Y = 0.0843 X$

Where Y is absorbance and 0.0843 is the slope of the line.

The absorbance of the unknown concentration of Kolosh solution $Y = 0.420$

Substituting Y value in equation: $Y = 0.0843 X$

$$X = 0.420 / 0.0843 = 4.98$$

In figure (3), Y is multiplied by 10^{-4}

* Y = 4.98×10^{-4} g / mL, the concentration of iron in one mL of Kolosh solution

The concentration of iron in 250 mL solution = $(250/1) * 4.98 \times 10^{-4} = 1245 \times 10^{-4}$ g

Percentage of Iron in Kolosh mineral = $[(1245 \times 10^{-4} * 100) / 5] \approx 2.5\%$

G - Qualitative Analysis of Elements

Experiments of analysis have been carried out with a view to obtain optimum results for qualitative determination of some elements that are present in measurable concentration and qualitative determination of other elements, which are present in trace or negligible concentrations in Kolosh mineral, that was due to lack of sophisticated equipment in our department. Table (3) shows the percent of some measure elements were represented in Kolosh mineral.

Samples of Kolosh mineral were digested and extracted in concentrated hydrochloric, nitric and sulphuric acids. The extracted solutions then reacted with different reagent for qualitative identification.

G-1- For group one elements in qualitative analysis (i.e. Ag, Hg and Pb elements) ^[15], post the reaction of Kolosh mineral with hydrochloric acid, all Ag, Hg and Pb compounds present in the mineral were converted to insoluble chlorides present in chlorinated Kolosh residue. To convert those chlorides to soluble nitrates, concentrated nitric acid was added to the residue and left for overnight. The mixture was filtered, and tests for presence of Ag, Hg and Pb in

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the filtrate were carried out. The tests showed positive result for presence of only lead. Figure (4) shows crystals of lead chloride under microscope on glass slid.

G-2- For copper group (IIA) elements in qualitative analysis (i.e. Hg, Pb, Bi, Cu, Cd, and As), and the arsenic group (IIB) (i.e. As, Sb, and Sn), tests showed no positive results for these groups elements. This may due to either the trace present of the elements that our reagent did not detect or total absence of elements.

G-3-For iron group (IIIA) elements in qualitative analysis (i.e. Fe, Al, Cr and Mn), test showed positive results just for Fe and Al. All filtrates post to the reaction of Kolosh mineral with different concentrated acids confirm the presence of iron (III) ion. This is in accord with the simple physical test that indicates the presence of Fe_2O_3



Figure (4) Lead chloride crystals under microscope on glass slid

G-4- For zinc group (IIIB) elements in qualitative analysis (i.e. Co, Ni, Mn and Zn) test showed negative results for all elements in the group. All filtrates post to the reaction of Kolosh mineral with different concentrated acids give negative testes for most transition metal ion. The absence of characteristic colors of transition metal ions in solutions beside the negative tests results confirm the absence of most transition metal ion.

G-5- For group (IV) elements in qualitative analysis (i.e. Ba, Sr, and Ca) test showed positive results just for Ba and Ca

G-6- For alkali group (V) elements in qualitative analysis (i.e. Mg, Na, K, and NH_4^+) test showed positive results just For Na.

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Results

The results of Kolosh samples reaction with different concentrated acids are summarized in table (1).

Table (1) shows the loss in weight of Kolosh mineral after its reactions with different concentrated acids

Acid Conc.	Weight of Kolosh/g	Weight of Precipitate/g	Weight loss /g	Weight Loss %
HCl	3	1.54	1.46	48.7%
HNO ₃	3	1.67	1.33	44.3%
H ₂ SO ₄	3	1.73	1.27	42.3%

The equivalent weights of NaF/g to the weight of sulphureted Kolosh sample /g are summarized in table (2).

Table (2): Shows the equivalent weights of NaF/g to the weight of sulphureted Kolosh sample /g

Weight of NaF/(g)	Weight of sulphureted Kolosh/ (g)
0.3	0.36
0.5	0.465
1	0.729
1.3	0.88
1.5	0.88
2	0.87

A plot of Kolosh sample weight /g versus sodium fluoride weight/ g shows that 1.3 g of NaF was enough to equate and react with all silica present in the sample.

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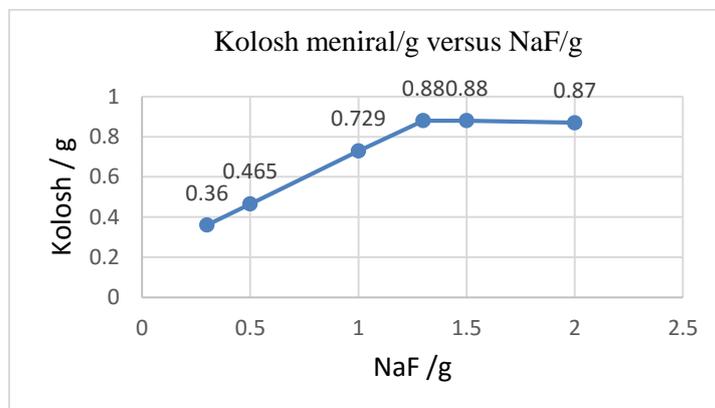


Fig. (1) Shows the weight of sulphureted Kolosh sample /g versus sodium florid weight /g

The determination of total organic carbon (TOC) in Kolosh sample shows change in sample color before and after heating the sample as shown in figure (2). The sample contains (5.3%) TOC.



Fig. (2) Kolosh precipitates colors before heating (A), after heating for Six hours (B)

The absorbance of Fe^{3+} colored solution in different concentrations that result from its reactions with potassium thiocyanate shown in table (3)

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Table (3) shows the different concentrations of standard and unknown of colored complex $[\text{Fe}(\text{NCS})(\text{H}_2\text{O})_5]^{2+}$ solutions versus absorption at $\lambda = 460\text{nm}$.

Concentration $\times 10^{-4}$ g/mL	Abs. at $\lambda = 460$ nm / standard	Abs. at $\lambda = 460$ nm / unknown
0.0	0.000	-
1.6	0.133	-
3.2	0.265	-
4.8	0.423	0.420
6.4	0.543	-
8.0	0.657	-
9.6	0.815	-

Figure (3) shows a plot of concentration of colored complex $[\text{Fe}(\text{NCS})(\text{H}_2\text{O})_5]^{2+}$ versus absorbance is straight line which emphasizes application of Beer's law

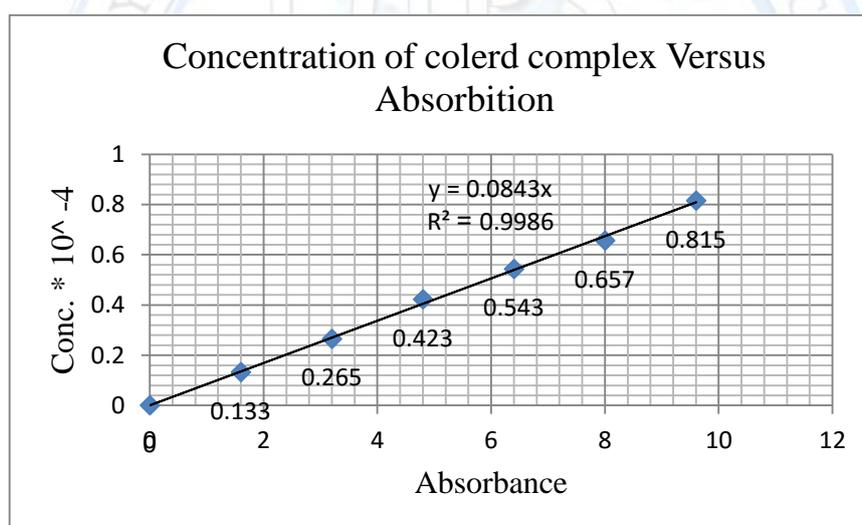


Figure (3): Plot of concentration of colored complex $[\text{Fe}(\text{NCS})(\text{H}_2\text{O})_5]^{2+}$ versus absorbance is straight line which emphasize application of Beer's law

Determination of total organic carbon (TOC) shows that the percentage of TOC in the sample = 5.3%, percentage of SiO_2 = 26.9%, and of iron = 2.5, as shown in tables (4).

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**Tables (4) show quantitative analysis for silica, TOC and iron as mentioned in aforesaid
3-D to 3-F sections.**

Element	Percentage %
Silica	26.9
Total organic carbon	5.3
Iron	2.5

Qualitative analysis of elements shows detectable concentration of Ca ion and very low presence of Pb, Al, Ba, and Na.

Discussion

Experiments have been carried out in different media with a view to obtain optimum results for extraction and qualitative determination of some elements present in very low concentration and qualitative determination of elements present in higher concentration in Kolosh formation mineral, due to the lack of sophisticated equipment.

A- Simple Test: The simple physical test based upon the ferromagnetic property of Fe^{3+} . Almost all Iron (III) oxides Fe_2O_3 are ferromagnetic, which means they all attract to magnets.

B- Missing Weights;

Table (1) shows different lost weights of Kolosh samples caused by its reaction with different acids. The lost weight was less for the reaction of sulphuric acid with Kolosh sample. The reason for that was, when sulphuric acid react with Kolosh sample, many elements present in the sample converted to insoluble or slightly soluble sulphates such as $CaSO_4$, $PbSO_4$ or $BaSO_4$. The insoluble new compounds remain in the residue where it increases the weight of the precipitate. As for the reaction with nitric acid, the missing weight was more than with sulphuric acid, but less than hydrochloric acid. This may be attributed to the formation of basic ferric nitrate which is insoluble or slightly soluble in dilute nitric acid or water. ^[16]

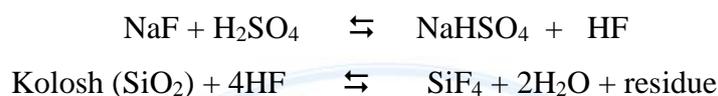
The reaction of the sample with hydrochloric acid yields largest lost in original weight of the sample. This is because all chlorides are more or less soluble in water.

C- The absence of hydrofluoric acid in our laboratory pushed to prepare it instantly by addition of excess concentrated sulphuric acid to the mixture of NaF with sulphurated Kolosh sample. Initially the reaction of NaF with H_2SO_4 conc. was started with liberation of some

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unreacted chunking white fumes of HF. The liberated HF within the mixture was immediately reacted with the available silica present around. The reaction was held in stainless steel container, as it had resistance to high concentrations of sulphuric or hydrofluoric acids. The reaction was left overnight to ensure the complete reactions of the component, according to the following chemical equations:



D- Before starting determination of total organic carbon (TOC), all inorganic carbon was rid of it by converting them to soluble chloride and nitrate. Determination of (TOC) needs high temperature to oxidize all organic carbon in the sample. This was provided by two naked hot torch flames placed beneath and above the sample. The naked flames helped and accelerated the oxidation of (TOC).

E- The percentage of iron was determined by KSCN because it had very low interference with other elements and high sensitivity toward Fe^{3+} ions. Table (3) shows that at concentration equal to $(4.8 \times 10^{-4} \text{ g/mL})$ the standard and unknown solutions had almost the same absorbance. According to Beer's law that means they had same concentration.

F- Existing Elements: Qualitative analysis of elements shows very low presence of Pb, Al, Ba, and Na.

Conclusion

This work reveals that Kolosh formation samples from nearby Hizop village near Kaniwatman area in Kurdistan region north of Iraq contains a reasonable percentage of silicon oxides, organic carbon and calcium, medium percentage of iron, very low detectable concentrations of aluminum, lead, barium and sodium. The absence of specific colored solutions extracted from the reactions of Kolosh samples with different acids reveals the absence of almost all transition metals within the samples. Moreover there were no any positive tests that confirm their presence in the mineral. The dark olive color of the mineral is caused by the presence of organic carbon. The lack of plants growth on the exposed areas of Kolosh formation may be caused by the presence of some toxic compound of elements in Kolosh formation affecting the living organisms.

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Clarification

We would like to point out that in this work we don't claim that we cover all elements that may present in Kolosh sample at Hizop village area, due to the lack of sophisticated equipment in our department. Besides we adapt our self's to overcome many obstacles that we faced through this work. We emphasize that this work is not in the field of geochemical branch of science.

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