Characterization of Black Shale of Chimiari Khyber Pakthunkhawa Region of Pakistan for Its Potential as Multi Minerals

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Abstract: ***The increasing demand has open the reach field to develop low grade ore deposits which were either technically impossible to develop or have serious environmental sequences or both. Black shale deposits are one of the low grade but of immense quantity reserves in the world. In Pakistan black shale deposits occur in Balakot, Kaghan Valley, and Jhelum Valley in AJK and Tarbela in Ghandgarh Range Khyber Pakthunkhawa (KP). The physical, chemical, mineralogical, radiometric and biological characterization of Chimiari black shale was carried out. The sample was ground to 72 mesh. The physical properties like color, streak, texture, moisture contents, hardness, specific gravity, voids ratio and porosity were studied. A low level γ-activity of 40 CPS was detected with the help of Macpher scintillation counter. In order to identify the minerals thin sections were studied and XRD was performed. The minerals identified are quartz, illite (a dioctahedral mica-mineral), K-feldspar, calcite and pyrite as the main minerals of black shale sample. Then the sample of black shale was chemically analyzed for SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, K_2O, SO_3, V_2O_5, MnO_2, TiO_2, Rb_2O, ZnO, CuO and C (Total) by XRF technique and comparison was made with natural average earth crust composition to see which minerals have been up graded. Finally the presence of some rod-shaped, motile and aerobic bacteria in the black shale ore sample was observed under the phase-contrast microscope at X100 magnification.***

Keywords: black shale, Chimiari, minerals, identification

1. Introduction

1.1 Black Shale Ore Deposits in Pakistan

In Pakistan, black shale deposits are widely distributed in Precambrian formation [1] in Khyber Pakthunkhawa (KP) formerly called North West Frontier Province (NWFP) and adjoining areas of Azad Jammu and Kashmir (AJK).

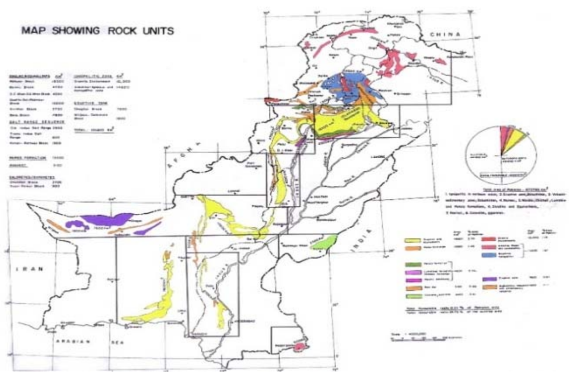

Figure 1.1: Black shale ore deposits in KP (Pakistan) and adjoining areas of Azad Jammu and Kashmir.

The general areas of occurrences of black shale deposits are Tarbela in Ghandgarh Range Khyber Pakthunkhawa (KP); and Balakot, Kaghan Valley and Jhelum Valley in AJK. These areas are shown in Figure-1.1.

These rocks are in the form of continuous and semi-continuous zones / beds, isolated lens, oiled bodies and of streaks hosted invariably in slate to phyllite to schistose lithologies.

The Precambrian formation hosting black shale deposits include:

- Salkhala Series in North Hazara Region and AJK
- Manki Formation in Ghandgarh Range with exposure near Nowshera and Tarbela Areas, and
- Alpuri Schists in Malakand Area

These black shale deposits of Pakistan have similar characteristics of geology and mineralogy with the Alum-Shale (Sweden) and Chattanooga Shale (USA) deposits [2]. The deposits of black shale in certain areas of Balakot, Kaghan Valley, Jhelum Valley in AJK and Tarbela in Ghandgarh Range (KP) also showing radioactivity [3]. A view of Chimiari black shale is shown in figure 1.2 and 1.3.

1.2 Chimiari Black Shale

1) Location

Chimiari black shale ore-deposit is one of the experimental black shale ore deposits, which exists near Tarbela about 11-Km south of Ghazi town (KP). The site is located on the northern slope of Ghandgarh Range near village Chimiari. Chimiari black shale deposit is located at Grid 775891 of Topo-sheet No. 43-C/9.

2) Flora and Fauna

The area is thickly covered with wild type shrubs (Sanatha) and small trees of Kahu, Pholahi and Kikar. Other trees growing along the nalah bank are banyan trees with some kind of olive and sheesham trees etc. Agricultural land is very less and scarce especially in the area in the vicinity of the site. Only some patches of land, which have been leveled and prepared by the local inhabitants by themselves to grow, some green fodder or any seasonal crop. Because of the high relief and unavailability of electricity, the water of the channel cannot be used for cultivation purposes and thus the agriculture is arid. The hills are mainly Shamlot forest, which is full of animal life including birds and animals of a number of species. However, no harmful animal except some snakes were seen [4, 5].

Figure 1.2*:* A scenic view of Chimiari black shale ore deposit site, Ghazi Tarbela, KP

3) Topography of Chimiari Site

The site is located on the northern slope of Ghandgarh Range near village Chamiari. The relief is high and the slopes are steep. The area is folded and faulted and a major fault called "Baghdarra Fault" is a prominent structural feature. There is a water channel continuously flowing due to influx of water from some natural springs and mountain water channels at base level of the site. On the southern edge of the water channel (nalah), there is a small rural settlement named "Dheri". The access to the site was by small pedestrian passages, which were developed over the years by the wood-cutters and some during the exploration activity to carry the rig parts at the drilling points. The area was inaccessible for jeeps and other vehicles. Therefore, pick-ups have to be parked some ½ km back from the zero-point.

Figure 1.3: Another scenic view of Chimiari black shale ore deposit site, Ghazi Tarbela.

4) Geology

Local geology of the area is generally dominated by the marine deep to shallow sequences of early to late Proterozoic age. The Ghandgarh Range is located in the southern Hazara area in the fold and thrust belt of the southern Himalaya in Pakistan. The area is folded and faulted and a major fault called Baghdarra Fault is a prominent structural feature. The tectonic movements have caused total destruction of the host rock along the upper contact. This phenomenon caused sub-aerial weathering process in the host rock after uplifting of the area.

On the very foot of the site, there is a mountain channel, which continuously flows due to influx of water from some natural springs. On the southern edge of the nalah, there is a small rural settlement named Dheri. The black shale formation is about 10-km long both in the northeast and southwest directions and up to 200-meters thick mineralized zone [4, 5].

Figure.1.4: Closer view of surface zone of Chimiari black shale

As a result of weathering process, minerals of the host rock were partially leached out downward direction along with rainy percolating water through fractures and subsequently, these were trapped immediately below the present ground level. So, the black shale deposit strata have been divided into three zones, surface zone is shown in figure 1.4.

5) Near Surface Zone / Oxidized or Weathered Zone

Black shale on the surface is very much sheared and the rock contains incoherent pebbles and black soil at some places looking like alluvium deposited on the flat lying areas along the slope. Thickness of this zone is 15-17 metes. Originally, pyrite was present in the shale–matrix, but it has been oxidized to sulfuric acid and ferric sulfate/ and leached out with the bacterial activity of indigenous iron- and sulfur-oxidizing bacteria (Acidithiobacillus ferrooxidans). At some places, the formation of iron oxide minerals (limonite, hematite and magnetite) exists at the surface (Figures-1.5 & 1.6). Figure 1.7 is showing the effect of water channel flow.

Figure 1.5: A view of near surface zone showing weathering/ xidation of pyrite due to air and bacteria (brown color indicating iron oxide minerals.

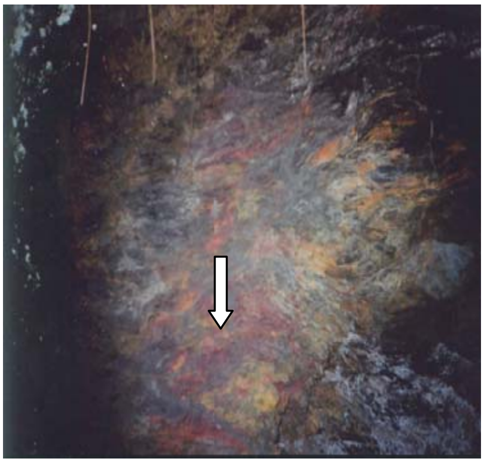

Figure 1.6: Natural bioleaching/ weathering of black shale ore at the near surface zone of Chimiari deposit site showing iron-oxide minerals.

Figure 1.7: Water channel flowing through the surface of weathered zone.

6) Zone of Concentration

This zone extends up to the depth of about 25 meters and contains fresh pyrite. Lithologically, black shale in this zone is fractured due to tectonic activity and the intensity of fracturing decreases towards the depth (Figure-1.8).

Figure.1.8: Fractured core sample of black shale taken from drill coring.

a) Primary Zone/Zone of Initial Fixation

The black shale is hard with slaty cleavage. Fresh, tiny and large crystals of pyrite are present along quartz veins can be seen in figure 1.9 below.

Figure 1.9: Black shale ore showing pyrite crystals as glittering in the anomalous zone (pyrite crystals are shown by arrows).

2. Methods and Materials

A fractured ore sample of 11kg was collected from the Chimiari black shale deposit. The sample was crushed in a Blake type Jaw crusher. The open & closed settings of the crusher were 1¼" and ½' respectively. The size of the sample was further reduced to 2.5mm with the help of roller crusher. The sample was then divided into eight equal parts by coning-quartering technique. One of part was ground in the Disc Pulverizer made by Mine & Smelter Supply Co. Denver USA. The final size was reduced to -70 mesh i.e. -212µm after sieving. The specific gravity of the core sample of Chimiari Black shale was measured by cylindrical method. A known weight of sample was placed in cylinder containing known volume of water. The practice was repeated thrice and the difference of volume was noted each time. Gamma activity of grinded black shale sample was carried out with the help of Macpher scintillation counter.

For mineralogical analysis XRD technique was used. For this purpose topfill powder mounts were used with CuKα-radiation and a vertical, wide-range goniometer (D8 Discover, Bruker AXS, Germany) equipped with a diffracted-beam monochromator and a Θ compensating slit. Elemental analysis of black shale ore sample for carbon, nitrogen and hydrogen contents was carried out on Elemental Analyzer EA 1108.

3. Results and Discussions

B. Physical Characteristics of Chimiari Black Shale

1) Sample Color and Streak

Black shale is a dark-colored mudrock [6]. The sample is of to dark grey to black color which is due to presence of carbon both graphitic and organic. A grey colored metallic oily layer was found on the surface of water. The streak was of dark color. The sample of black shale was found insoluble in water however a grey colored metallic oily layer was found on the surface of water. The Chimiari black shale is alkaline in nature having pH equal to 7.3. The organic material that gives black shale its distinctive characteristics is derived from living things [6]. The coloring material in individual shales might be carbon, hydrocarbon or finally divided iron sulfide [7, 8].

2) Texture

Black shale contains organic matter and silt and clay-size mineral grains that accumulated together [6]. The sample was a very fine grained with mineral grain invisible except under a microscope. It is finely laminated and splits easily along the bedding planes. Flakes / lamination are visible and can be easily separated which is characteristic of black shale. In the fractured core sample pyrites can be easily seen with naked eye. Below is a photomicrograph of Chimiari black sample.

Figure 3.1: Photomicrographs of Chimiari black shale

3) Moisture Contents

Following procedure was performed to measure the moisture content:

1. About 1kg of size (-8 mesh) black shale was sampled. The material was finely ground (-212μm) in a pulverizer.
2. One gram of ground black shale was taken in a pre-dried and weighed crucible.
3. The crucible along with the sample was transferred to furnace and heated at 110° C for one hour.
4. After one hour the crucible was shifted to desiccators and was allowed to cold down to room temperature i.e. 30°C
5. The crucible was weighed and the difference of weight was noted. The procedure was performed thrice.
6. The average moisture content was found 0.9%.

4) Hardness

The hardness of the sample was measured with Moh's scale. Although the sample was soft touch and leaving its blackish color when touching with hand but it cannot be scratched by nail. It was easily scratched by knife and hardly by coin. So hardness of the core sample lies between 4 and 5.

5) Specific Gravity

The black shales have specific gravity of 2.1 as calculated from drill. It was found that the average specific gravity of the core black shale was 2.85 which is almost similar to already reported for black shale. [9]

6) Void Ratio and Porosity

The porosity of black shales varies from 0 to 50% depending upon depth and level of compaction. Usually shales are highly porous but have low permeability. Permeability is a measure of a material's ability to transmit fluids. It is low in shales and is typically between 0.1 to 0.00001 millidarcy (md). The porosity of core samples of black shale taken from the Marcellus in one well in New York is ranged from 0 to 18% [10].

The sample was got tested for void ratio and porosity from SOILCON laboratory Lahore. The loose sample has void ratio 0.82 and that of eroded has 0.93. Porosity is found 45%.

C. Gamma (γ)-Ray Spectrometric Analysis of Black Shale Sample

Black shale typically contains trace levels of naturally occurring radioactive materials/metals contents (NORM) such as uranium-238 and radium-226 at higher levels than surrounding rock formations [9]. The γ-activity of natural ^{235}U present in the shale sample was noted to be 40 counts per seconds (CPS). The back ground activity was found 35 CPS for the same. The higher γ-activity in the sample than that of the normal or back ground means that some radio active metals may be present.

The technical data obtained from the surface- and subsurface radiometry (γ-logging) shallow excavations (trenching and aditing), ROAC survey, Pb-isotopic studies, shallow and deep drilling has suggested that the Chimiari black shale deposit is rich in uranium like [7] but is lacking of any mineralization in the form of low or medium grade uranium ore deposit.

Table 1: Gamma (γ)-ray Spectrometric analysis of low-grade Chamiari uraniferous black shale ore

Radio-isotope	Activity (Bq kg^{-1})
^{235}U	97.6 ± 1.34
^{226}Ra	920.10 ± 0.31
^{228}Ra	178.90 ± 2.58
^{40}K	518.10 ± 15.10

Gamma-ray spectrometric analysis of black shale ore sample is shown in Table-1.1. The γ-activity of natural ^{235}U present in the shale sample was observed as 97.6± 1.34 Bq Kg-1 of sample. The γ-activity of a uranium sample comes from ^{226}Ra and its decay products, particularly ^{214}Pb and ^{214}Bi. The γ-activity of ^{226}Ra, ^{228}Ra and ^{40}K were also detected in the sample.

D. Mineralogical Characteristics of Black Shale

1) Thin Section Studies

Four thin sections of the fractured core sample were prepared. The section under micro scope did not show the presence of any particular mineral. However, thin sections had shown the brightness, darkness and cluster of colorful spotting. The cluster of colorful spotting represents the limestone that has been adsorbed on the layering of quartz particles. The black portion also did not show any type of mineral. The darkness may be mainly due to the presence of organic matter or carbon content which is peculiar of black shales.

Individual minerals grains were not visible in a thin section which means that the mineral grains are of very fine size. The metal contents of black shale may be hosted in fine-grained quartz, calcite and carbon.

2) X-Ray Diffraction (XRD) Analysis

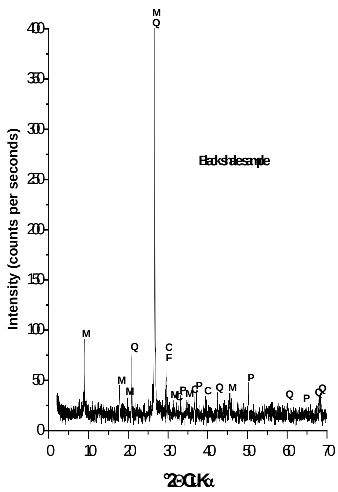

Figure 3.2: XRD diffractogram of untreated Chimiari black shale sample. Symbol designations: C = calcite, F = Feldspar, M = mica (illite), P = pyrite, Q = quartz

All samples were scanned from 3 to 70 °2Θ in increments of 0.04 °2Θ with a 4-sec counting time. X-ray diffractogram matching was performed in the automatic mode against JCPDS powder diffraction library, then manually to confirm the automated identifications of phases.

3) Minerals Identification

The main minerals identified by the XRD data are reported in Table-2. The minerals identified are quartz, illite (a dioctahedral mica-mineral), K-feldspar, calcite and pyrite as the main minerals of black shale sample.

The mineral identification with XRD shows the minerals present are of clay minerals. The up-gradation technique may require fine size grinding to remove quartz, calcite and organic carbon.

Table 2: *Mineral analysis of Chimiari Black Shale*

Mineral identified	Mineral Formula
Quartz	SiO_2
Illite	$(K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4O_{10}[(OH)_2,(H_2O)]$
Pyrite	FeS_2
Calcite	$CaCO_3$
Feldspar	$KAlSi_3O_8$

E. Chemical Analysis of Black Shale

The chemical analysis for carbon, nitrogen and hydrogen was carried out on Elemental Analyzer EA 1108 and by the metal Electron dispersive spectroscopic (EXD) and by standard spectrophotometric method as described by Johnson and Florence (1971) i.e. X-Ray Fluorescence (XRF).Since the γ-activity was present in the sample so the sample was analyzed for radioactive Uranium metal. The analysis was carried out by titration method. The average contents of Uranium metal were 45ppm. The Electron dispersive spectroscopic (EXD) results showed that Rb, ZnO and CuO are also present in the sample in minute quantity and their concentrations are 0.015, 0.03% and 0.013% respectively.

1) Analysis for Carbon Nitrogen and Hydrogen

The results are presented in Table-3.

Table 3: *Elemental analysis of low-grade Chimiari black shale*

Element	Results (%)
Carbon	18.0-22.0 ± 1.20
Nitrogen	1.1- 1.45 ± 0.05
Hydrogen	2.0-3.1 ± 0.11

Table 3 showed the carbon, nitrogen and hydrogen contents in the black shale ore sample indicate the presence of some organic compounds (hydrocarbon compounds) as plant material [2] results are much similar to already reported [11]. The black shale horizon is rich in OM. The chemical analysis of sample tabulated in Table 4 confirmed that shale is enriched in organic carbon. The results showed that TOC 17.92% is the 2nd major element after silica.

2) Chemical Analysis of Black Shale for Metals

The presences of other metals like Co, Mo, Ni, Ta, W and U; Rare-earth elements viz., Ce, Gd, Dy, Nb and Y were also detected in Chimari black shale. The presence of gold (Au) was as precious metal was also detected in the near surface shale samples [2]. These results need further reconfirmation of the analytical data.

The prepared sample was analyzed for Si, Al, Fe, Ca, K, S, Li, V Mn, Ti, Rb, Cu, Zn, Total Organic content (TOC). The Chemical analysis was carried out by standard spectrophotometric method as described by Johnson and Florence (1971) i.e. X-Ray Fluorescence (XRF) results were the reported in Table-4.

***Table 4:** Chemical analysis of Chimiari black shale sample*

ELEMENT	RESULTS (%)
SiO_2	49.86
Al_2O_3	11.6
Fe_2O_3	7.43
CaO	5.18
MgO	0.56
K_2O	2.55
SO_3	4.12
V_2O_5	0.26
MnO_2	0.14
TiO_2	0.81
Rb_2O	NR*
ZnO	NR*
CuO	NR*
C (Total)	17.92

*N.R. means Not Reported Results due very small concentrations.

The concentrations of V and Ti are much higher than that of U. The sample could not be detected for Rb_2O, ZnO and CuO by XRF technique. However analysis by Electron dispersive electronic technique (EDX) technique shows the presence of these elements. The concentrations of Rb_2O, ZnO and CuO by EDX method is 0.014%, 0.017% and 0.012%.

3) Gangue Minerals

The chemical analysis showed the high contents of quartz (49.86%), alumina (11.60 %), iron (7.43%), calcium (5.18%), and sulfur (4.12%). SiO_2 are the major impurities which is present mainly due to quartz minerals. The second major impurity is Al_2O_3 which is associated with clay minerals. K_2O is also associated with clay minerals. Calcium occurs in the form of calcite.

F. Metal Distribution and Grade

The economic grade of black shale may not be determined on the basis of any of the individual metal that has been concentrated as mentioned above because no individual metals is in such a higher concentration that it may be recovered economically. A comparison of ubiquitous oxide / elements in Chimiari black shale and in earth crust is shown in Table-5. The comparison showed that the shale sample under study is slightly enriched in manganese (Mn) & Titanium (Ti) as these are enriched by a factor of 1.4 and 1.5 respectively as compared to their average concentration in earth crust [12]. The shale is moderately improved in vanadium (V) & uranium (U) by a factor of 21.58 and 16.67 respectively. The sample was found highly rich in organic carbon because the factor for enrichment is 896 times.

Table-5: Comparison of contents of ubiquitous oxide/elements in Chimiari black shale and in Earth crust

Element	Abundance as oxide/element at Chamiari		Abundance in Earth Crust as oxide/element [12]		Relative enrichment at Chamiari
	%	ppm	%	ppm	
SiO_2	49.86		28.20		1.77
Al_2O_3	11.60		8.23		1.41
Fe_2O_3	7.53		5.63		1.34
CaO	5.18		4.15		1.25
MgO	0.56		2.33		0.24
K_2O	2.55		2.09		1.22
SO_3	4.12		0.88		4.71
C	17.92		0.02		896.00
TiO_2		8140.0		5700.0	1.43
MnO_2		1410.0		1000.0	1.41
U		45.0		2.70	16.67
V		2590.0		120.0	21.58
Rb		140.0		90.0	1.56
Zn		170.0		70.0	2.43
Cu		120.0		60.0	2.00

The presence of higher amount of value able metals like Vanadium and Uranium in the sample shows that these have higher concentration than that of normal in shale which means that these been concentrated in Chimiari black shale. Therefore the Chimiari black shale may be graded as poly metallic type shale for C, Mn, V, Ti and U.

The grade of the un oxidized fractured zone of Chimiari black shale will thus be mainly determined on the basis of metal contents of C Mn, Ti, V & U and will also on the type of technology/process that will be developed to recover each of the metal. The enrichment factor for Ti and Mn will also contribute to determine the economic grade. The chemical analysis showed the presence of radioactive U-metal in very small amounts i.e. 0.0045% (45ppm). It means that the Uranium (U) has been enriched by a factor of 11.67 as compared to its abundance in earth crust. The relative enrichment factor of carbon is 896 which are attracting Since EDX technique showed minor concentrations of Rb, Cu and Zn. their recoveries as by product may also be considered.

G. Microbiological Characterization of Chimiari Black Shale

The presence of some rod-shaped, motile and aerobic bacteria in the black shale ore sample was observed under the phase-contrast microscope at X100 magnification (Figure-3.3). Standard microbiological techniques were employed for the isolation and characterization of indigenous bacteria present in the black shale ore sample.

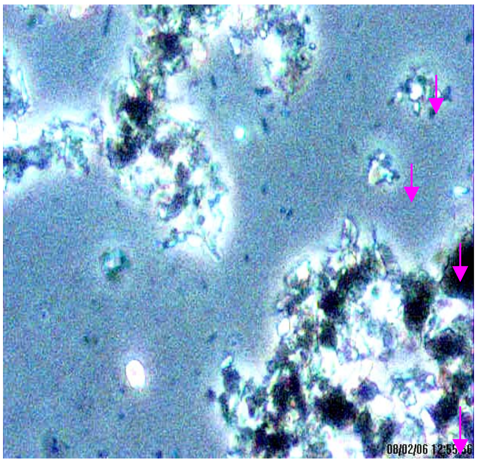

Figure 3.3 Phase contrast microscope (X100 magnifications) showing the presence of rod-shaped bacteria (indicated by arrow) in black shale ore-water slurry.

Indigenous microflora of acidophilic iron- and sulfur-oxidizing mesophile (grow at 10-45°C) and moderate thermophile (45 - 60°C) were isolated and characterized as Acidithiobacillus ferooxidans (BSTF-1) similar as [13] and Acidithiobacillus caldus (BSMT-1), respectively.

4. Conclusions

The sample from Chimiari black shale deposit was examined & studied for the physical, mineralogical, chemical, radiological and biological characterization. The followings are the conclusions drawn from the studies mentioned above.

The flakes are easily visible and pyrite can also be seen with naked eye. The streak is black. According to the Moh's scale the hardness is between 4 & 5. The specific gravity is 2.85 and moisture contents are 0.9%. The porosity is found 45%. The sample shows low level γ-radio activity. The black shale, under study, is a complex in nature.

XRD studies show that quartz, K-feldspar, calcite, illiminite and pyrite are present in the black shale of Chimiari. Table-5 shows that the elements of C, Ti, Mn, U and V have been accumulated as these have attained an enrichment level of 896, 1.43, 1.41, 16.67 and 21.58 respectively. Therefore the Chimiari black shale may be characterized as polymetallic / polyelement black shale because of the enrichment of C, Ti, Mn, U and V. TOC is the 2nd highest element in concentration and its concentration is 17.92%. It is therefore concluded that Chimiari black shale is heavily enriched with TOC.

Rod shaped bacteria are found under the phase-contrast microscope at X100 magnification. This means that the bio-froth flotation is possible.

5. Recommendations

In the light of the conclusions, it is recommended to carry out the following studies on Chimiari black shale deposit.

A. Detail analysis of Chimiari black shale for trace element such as Ag, Au, Li, Se, etc. should be carried out. So that these may also contribute towards the economic recovery of valuable metals.
B. Since the black shale is a complex in nature for recovering valuable minerals, so the detailed study of surface properties of minerals is required to sufficient utilization.
C. SiO_2 being the main impurity, could not be separated from valuable metals. Therefore it is strongly recommended that flotation experiments with "cation" class of reagents may carried out as these reagents are characterized by their marked selectivity towards silicates.
D. Fine grinding may be successful to segregate the valuable mineral contents and organic carbon content but with pretreatment procedures.
E. Detailed study for the respective minerals should be carried to get the mesh of liberation for each of the mineral present.
F. Direct leaching with H_2SO_4, HF and H_2O_2 as oxidizing agent may be utilized for vanadium and Titanium extraction.
G. Environmental studies regarding the concentration of some toxic elements are carried out for better understanding of the mineral deposit.

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