



Geochemistry and Mineralogy of Soil in Baiji Oil Refinery, and Surrounding Area.

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ABSTRACT

This study aims to demonstrate the relationship between the major oxides and mineralogy, as well as the effect of clay minerals in increasing the distribution of heavy metals in the soil of present study.

22 samples were collected from the refinery and nearby area. All samples were analyzed to determine the concentrations of heavy metals by atomic absorption spectrophotometer (AAS). The results showed that the concentration of heavy metals (Cd, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) the three samples with the highest concentration of heavy metals and the three lowest samples were identified to conduct an analysis of the major oxides using an XRF. The results showed the highest percentage of silica oxide and calcium oxide in comparison. Other oxides such as (Al₂O₃, Fe₂O₃, CaO, MgO, Na₂O, K₂O, SO₃, and LOI). The results of clay and non-clay minerals, which were conducted for the three samples with the highest concentration of heavy metals, as well as for the three samples with the lowest concentration of heavy metals, using an XRD technique, showed that most common non-clay minerals were Quartz, gypsum and calcite. Non-clay minerals with lower percentages such Albite, Dolomite, Halite, Rutile, small percentages of the clay minerals were represented by chlorite, Palygorskite and Illite.

The results of the study showed that an increase in silica oxide indicates the presence of quartz, and an increase in calcium oxide indicates the presence of carbonate minerals, especially calcite and dolomite.

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1. INTRODUCTION

The majority of Iraq is covered with Quaternary sediments, which also make up the majority of its soils. The area consists of recent floodplain sediments (Holocene) from the Tigris and Euphrates rivers and their tributaries, as well as sediments carried by wind, primarily in the form of sand dunes that are found in the northern and northwestern regions of the sedimentary plain [1] The majority of these deposits mostly comprise clays, mud, and sand, along with chemical deposits of carbonates. [2] Conducted a comprehensive analysis of the mineral composition of soils in the northern and central regions of Iraq. The authors highlighted the mineralogical characteristics of soils and minerals found in flood sediments and river terraces, as well as the variations in mineral composition at different soil depths. It was discovered that montmorillonite is the predominant mineral, along with other minerals present in varying amounts.

This study was conducted in the soil of the Baiji oil refinery and surrounding region. Baiji refinery is situated on-site in Salahuddin Governorate (130 km north of Baghdad), in middle the road between Baghdad and Mosul. The area of study location lies in the unstable shelf within the Mesopotamian basin [3]. From a geographical perspective, it is located to the north of Baiji City, with specific coordinates in terms of longitude (43' 23" – 43' 33") and latitude (34' 52" – 35' 50"), Figure 1. This study aims to demonstrate the relationship

between the major oxides and clay minerals, as well as the effect of clay minerals on increasing the distribution of heavy metals in the soil.

2. METHOD

2.1 Atomic Absorption Spectrophotometer (AAS)

A total of twenty-two soil samples have been collected, with six taken from within the refinery and sixteen from regions outside the refinery. These samples were analyzed using geochemical analysis to identify the percentages of heavy metals, including (Cd, Cr, Cu, Fe, Mn, Ni, Pb, and Zn). The analysis was performed at the Center of Market Research and Consumer Protection on Baghdad University.

2.2 X-ray Diffraction

Out of the soil samples collected, six were selected for analysis. The three samples with the highest content of heavy metals (S1, S2, and S3) were chosen, as well as three samples with the lowest content of heavy metals (S13, S14, and S16), were selected with depth (5- 30 cm), chemical analysis was conducted in German-Iraqi Laboratory at the Department of Geology College of Science, University of Baghdad. This is to know the percentages of major oxides in study area.

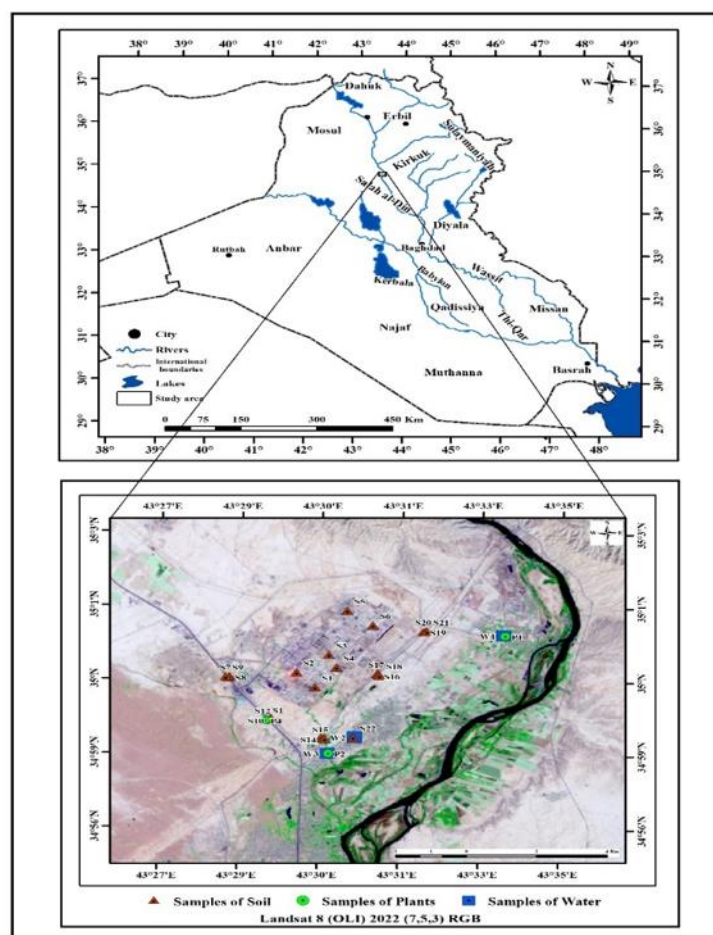


Figure 1. Location map of soil samples (Landsat8 (OLI) 2022(7,5,3) RGB).

2.3 XRD analysis

Clay minerals were diagnosed for six soil samples highest three soil samples in heavy metals content (S1, S2 and S3) and the lowest three soils samples in heavy metals content (S13, S14 and S16) to be Examined by XRD to realize the type and ratio of clay minerals. The selected samples were analyzed using the XRD technique (type Shiamatzu XRD-6000) at University Baghdad- College of science- Department of Geology. To identify clay and non-clay minerals in study area.

3. RESULTS AND DISCUSSION

3.1 Heavy metals

The concentrations of heavy metals in the investigated soils Table 1, indicate a clear influence of anthropogenic activities associated with the Baiji Oil Refinery. Cadmium (Cd) exhibited consistently high concentrations (36- 46 ppm; mean = 40 ppm), greatly exceeding natural background levels, suggesting continuous atmospheric deposition from petroleum refining processes, fuel combustion, lubricating oils, and industrial emissions [4],[5]. Chromium (Cr) and nickel (Ni) also showed elevated mean concentrations of 160 and 81 ppm, respectively, with the highest values recorded in the samples closest to the refinery and a gradual decline toward the more distant sites, indicating that refinery emissions and the combustion of crude oil are the primary sources of these elements [5],[6]. Nickel is particularly recognized as a characteristic tracer of petroleum because crude oil naturally contains Ni-bearing porphyrins, while chromium is commonly associated with catalysts, corrosion-resistant alloys, and industrial dust generated during refinery operations [4],[5]. Copper (Cu) displayed remarkably high concentrations (mean = 253 ppm), reflecting substantial anthropogenic enrichment that may be attributed to the corrosion of pipelines and metallic infrastructure, lubricating oils, industrial machinery, and maintenance activities within the refinery [4],[5]. In contrast, lead (Pb) showed moderate concentrations (mean = 25 ppm), which are close to typical global soil background values, although slight enrichment in several locations may be related to long-term atmospheric deposition, vehicular traffic, and historical industrial emissions [5],[6]. Manganese (Mn) and iron (Fe) exhibited concentrations within the expected natural ranges for soils, suggesting that their distribution is mainly controlled by the mineralogical composition of the parent material and weathering processes rather than industrial contamination [4],[5]. Zinc (Zn) showed generally low concentrations, with only a few localized enrichments, indicating that Zn contamination is limited and probably linked to specific point sources such as equipment wear or galvanized materials [4],[5].

Overall, the spatial distribution and enrichment patterns of Cd, Cr, Ni, and Cu demonstrate that petroleum refining activities are the dominant source of heavy metal accumulation in the adjacent soils, whereas Fe and Mn are predominantly of lithogenic origin and Zn reflects only localized anthropogenic inputs [7].

Table 1. Heavy metals concentrations (ppm) in the soil Baiji oil refinery.

Sample ID	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Zn
S1	44	437	51	25959	378	187	11	10
S2	46	410	56	16524	311	176	10	4
S3	45	322	56	27659	391	155	9	53
S4	43	296	61	18462	178	126	7	19
S5	42	326	160	18691	255	135	20	1
S6	43	151	264	15427	221	79	13	6
S7	40	166	101	25685	378	95	21	4
S8	41	135	160	21146	290	89	21	7
S9	40	116	175	23566	347	87	23	78
S10	42	97	135	29043	159	76	22	2
S11	40	97	345	10295	95	43	30	2
S12	37	124	256	17178	186	62	30	0.4
S13	36	78	360	13543	141	39	27	0.05
S14	40	71	367	11691	114	38	35	2
S15	36	82	71	17156	255	40	30	0.06
S16	39	86	456	8975	88	41	29	0.98
S17	38	78	478	10037	106	48	32	3
S18	40	86	382	15753	210	77	35	4
S19	38	112	404	13962	227	57	40	0.97
S20	37	82	456	10875	112	62	34	0.11
S21	38	78	390	12672	111	43	40	0.09
S22	40	93	375	16714	209	43	33	4
Mean	40	160	253	17319	216	81	25	9

3.2 Major oxides

The concentrations of the following main oxides are (Al₂O₃, Fe₂O₃, CaO, MgO, Na₂O, K₂O, SO₃, and LOI) were analyzed in the soil samples.

3.2.1 Aluminum oxide (Al₂O₃)

Aluminum oxide ranged between (6.6- 10.1%) with an average of (8.5%) in the soil of Baiji area, these values are below the global average for UUC and PAAS. the concentrations of this study are very closely to the average value of Al_2O_3 in soil of Baiji area according to [8]. Alumina is a common oxide in silicate minerals, that may be released by weathering [10]. [11] stated that aluminum is included in the composition of numerous minerals that are delivered through fluvial and Aeolian sources. Clay minerals are known to include conservative elements, and one of these elements is regarded to be a fundamental component [12]. Table 2, Figure 2.

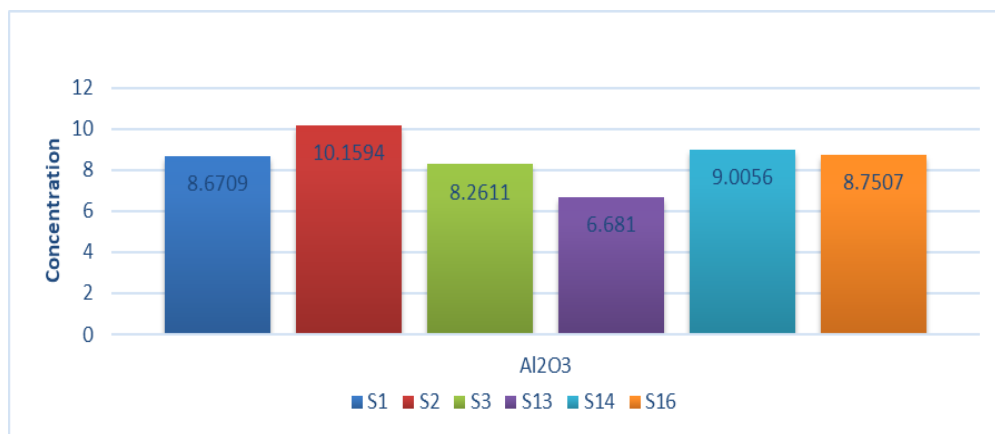


Figure 2. Distribution of Aluminum oxide (Al_2O_3)

3.2.2 Silica (SiO_2)

The concentration of SiO_2 in the studied soil ranged from (35.91-51.73%), with an average of (43.31 %). These values have a smaller magnitude compared to the global averages observed in soil for the Upper Continental Crust (UUC) and Post Archean American Shale (PAAS), Table 2. Silica the most abundant oxide in the study area, the significant abundance of silica is mostly attributed to its substantial occurrence in the form of quartz minerals and a lesser quantity of feldspar in the silt and sandy parts, as well as its incorporation into the composition of chemical of clay minerals. Silica increases compared to the other oxides due to its existence in the form of free silica (Quartz mineral), Figure 3.

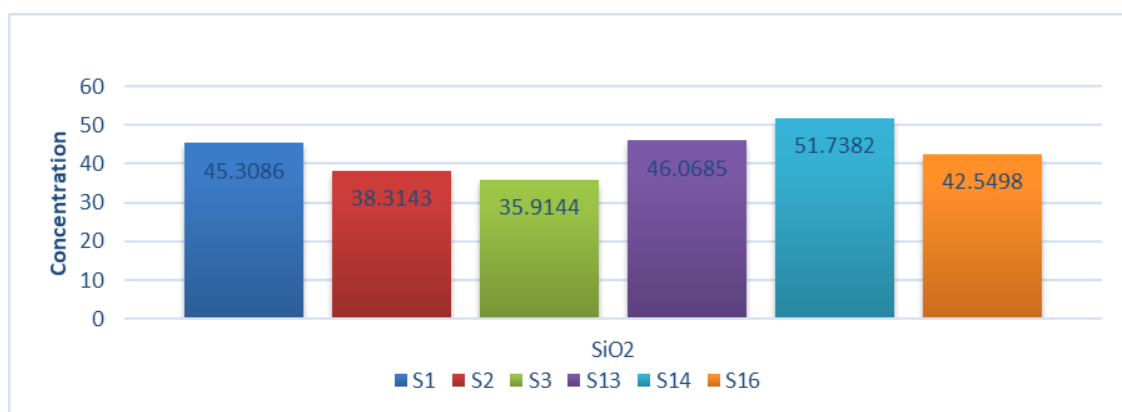


Figure 3. Distribution of Silicon oxides (SiO_2)

3.2.3 Iron oxide (Fe_2O_3)

It considers as significant catalysts in adsorption reactions. Fe_2O_3 is found in all soils and influence soil color, structure, and ion adsorption even at low concentrations [14]. It exists in soil as a cementing material or coating for mineral granules within the soil, iron oxides are associated degree of weathering, parent materials, and pedogenic accumulation or depletion processes [15]. The concentration of iron oxide in the studied area is ranging from (4.3-7.7%) with an average of (6.2%), This value is closer to global averages (UUC., PAAS.) Table 2. it is existing with most samples except (S3), it is also compared with the soil of Baiji area and was higher according to [8], Figure 4.

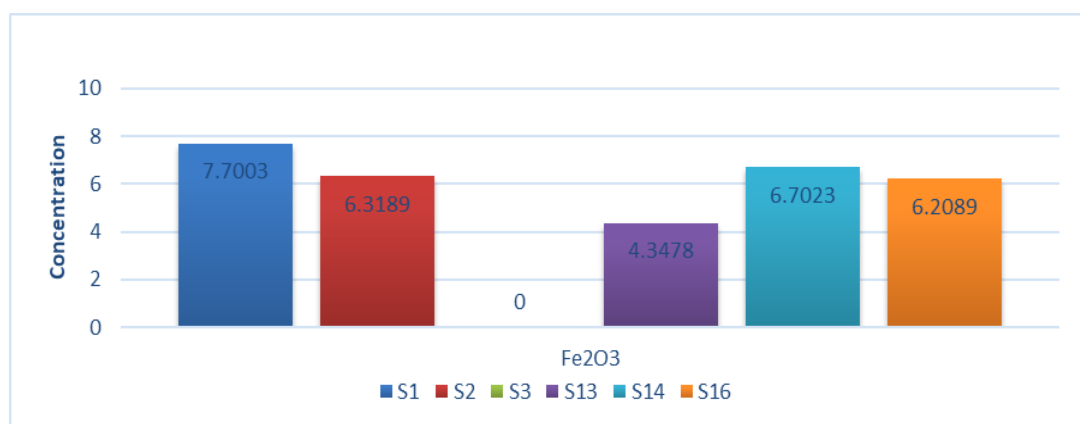


Figure 4. Distribution of Iron oxide (Fe_2O_3)

3.2.4 Calcium oxide (CaO)

The concentration of CaO in the studied area is ranging from (18.58-26.88%) with an average of (22.77%). The average concentration of CaO in the studied area is higher than in the Baiji area according to [8]. These values have a higher compared to the global averages observed in soil for the Upper Continental Crust (UUC) and Post Archean American Shale (PAAS), Table 2. The high content of CaO concentration in soil samples (S13, S1 and S16) (26.8, 25.3 and 23.8%) respectively indicate to a significant presence of carbonate minerals, specifically calcite and dolomite which is detected during x-ray analysis, Figure 5.

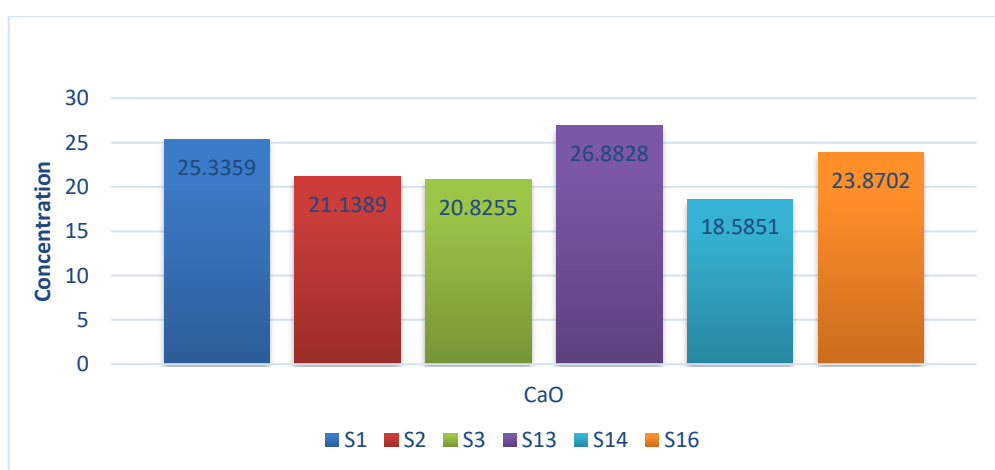


Figure 5. Distribution of Calcium oxide (CaO)

3.2.5 Magnesium oxide (MgO)

Magnesium oxide concentration in soil samples ranged from (2.8-6.6) % with an average of (5.3 %). The average value of MgO in the soils of the present study is higher than the average value of MgO in soil of Baiji area according to [8], these values are higher than global averages (UUC. and PAAS.), Table 2. Higher concentration is (5.1- 6.09%) in sample (S16, S1), respectively this is due to the existence of Dolomite mineral. Magnesium is present in the chemical structure of a number of clay minerals found in the study region, including palygorskite, illite, and chlorite, Figure 6.

3.2.6 Sodium oxide (Na_2O)

Sodium oxide concentration in the studied area ranged from (1.1-3.4%) with an average of (1.9%), these values are below the global average (UUC.) (3.9%) but higher the global values (PAAS.) (1.2%), average value of Na_2O is higher than the average value of Na_2O in the study area according to [8], Table 2. Sodium oxide is found in halite and clay minerals as exchangeable ions, Na_2O is a major component of clay minerals [13], Figure 7.

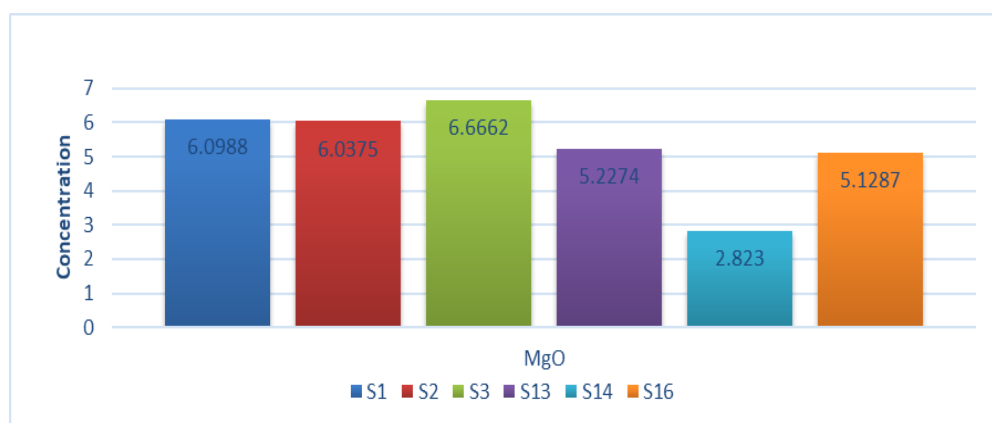


Figure 6. Distribution of Magnesium oxide (MgO)

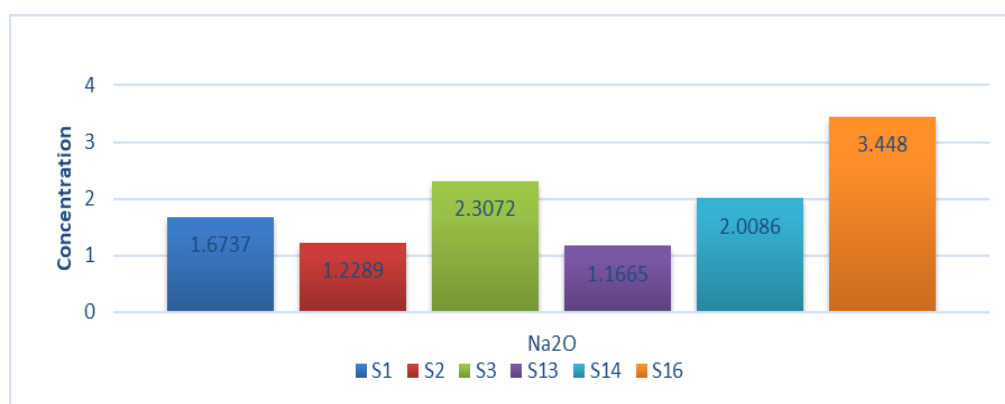


Figure 7. Distribution of Sodium oxide (Na₂O)

3.2.7 Potassium oxides (K₂O)

Potassium oxide concentration in the study area ranged from (1.4-2.4 %) with a mean of (1.8%), The average value of K₂O in the soils of the study area was slightly higher than the average value in soil of Baiji area according to [8] and below the global average for UUC and PAAS, Table 2. it is existing with most samples except (S3). Potassium existence is related to the occurrence of illite minerals, it found in clay minerals either as one of the exchangeable ions or on the surface of clay mineral layers [16], Figure 8.

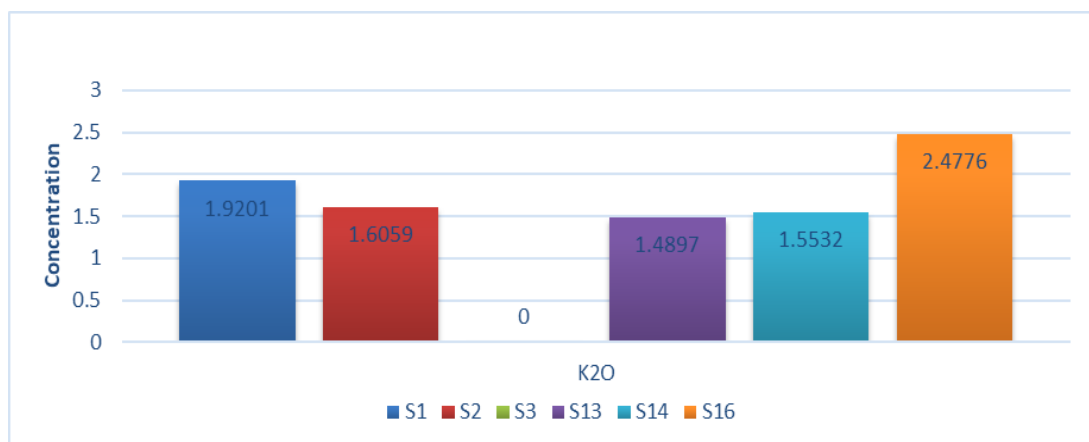


Figure 8. Distribution of Potassium oxide (K₂O)

3.2.8 Sulfur oxide (SO₃)

Sulfur oxide concentration is ranged between (18.4-2.1%) with an average of (9.1%) in the studied area Table 2. The higher value of SO₃ concentration in soil samples (S2, S3) (13.8-18.4%) respectively indicate a presence of oil residues. Sulfur oxide is found in (pyrite FeS₂), (chalcopyrite CuFeS₂), (galena PbS), and (sphalerite ZnS) [9], Figure 9.

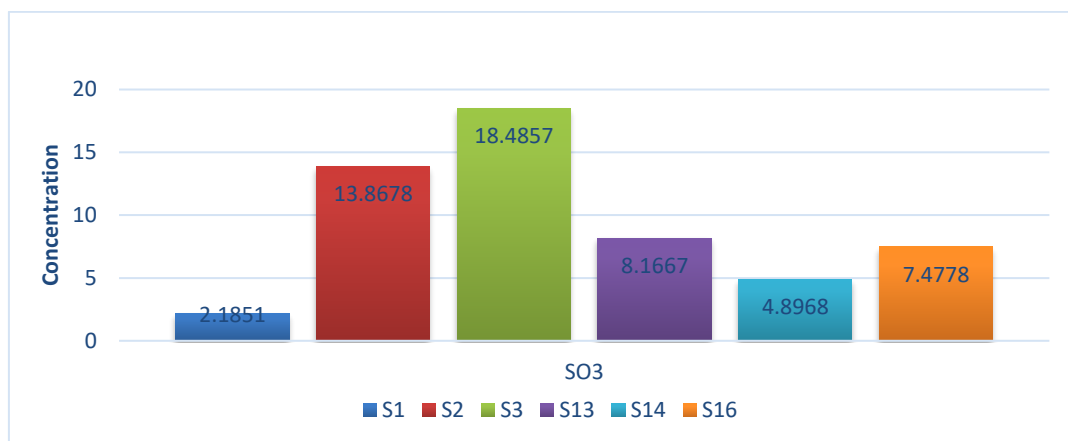


Figure 9. Distribution of sulfur oxide (SO₃)

3.2.9 Loss on ignition (L.O.I)

It restricted between (9.39- 19.64 %) with an average of (14.3%) Table 2. The most significant cause of loss on ignition as the vaporization of molecular water (H₂O⁺, OH⁻) present in the crystalline arrangement of clay minerals and the water adsorbed on their surfaces This process involves the disintegration of carbonate minerals and the subsequent emission of carbon dioxide, as well as the combustion of substances like sulphates (SO₃) and evaporated chlorine (Cl), and evaporate F [17],[18], Figure 10.

Table 2. Concentrations of major oxides (%) in the soils Baiji oil refinery and compared it with Global rates and previous study.

Sample ID	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	L.O.I.
S1	8.67	45.31	7.70	25.34	6.10	1.68	1.92	2.19	19.64
S2	10.16	38.31	6.32	21.14	6.04	1.23	1.61	13.87	12.2
S3	8.26	35.91	-	20.83	6.67	2.31	-	18.49	11.15
S13	6.68	46.07	4.35	26.88	5.23	1.17	1.49	8.167	14.28
S14	9.01	51.74	6.70	18.59	2.82	2.01	1.55	4.9	-
S16	8.75	42.55	6.21	23.88	5.13	3.45	2.48	7.48	-
Range	6.6- 10.1	35.9- 51.7	4.3- 6.7	18.5- 26.8	2.8- 6.6	1.1- 3.4	1.4- 2.4	2.1- 18.4	-
Average	8.59	43.32	6.26	22.77	5.330	1.97	1.81	9.18	5.45
[8]	8.11	-	3.86	14.43	3.68	1.38	1.34	-	-
P.A.A.S.	18.9	62.8	7.22	1.3	2.2	1.2	3.7	-	-
U.C.C.	15.2	66	5	4.2	2.2	3.9	3.4	-	-

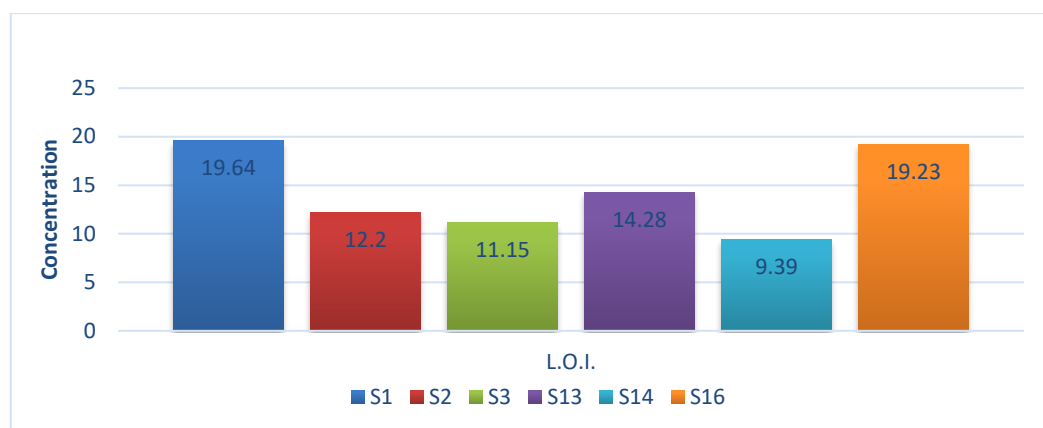


Figure 10. Results of LOI

3.3 Mineralogy

Six samples from the surface soil were selected, the high three soils samples with the concentration of trace elements (S1, S2, S3) and the low three soils samples with the concentration of trace elements (S13, S14, S16) to identify the non-clay minerals and clay minerals using XRD technique.

3.3.1 Non clay minerals

The XRD analysis indicated the existence of non-clay minerals including Calcite, Dolomite, Quartz, Gypsum, Albite, and Barite, Figures 11, 12, 13, 14, 15 and 16.

3.3.1.1 Carbonate minerals

The carbonate minerals diagnosed were calcite (CaCO_3) and dolomite $\text{CaMg}(\text{CO}_3)_2$. According to the XRD results, is the most abundant non-clay mineral in all selected soil samples contain calcite in the study area, with an average (13.8 %), Table 3. Calcite was identified at the basal reflection (3.03 Å), (2.09 Å) and (3.86Å) at an angle of (23.04°), (29.43°), (47.13°) 2θ , respectively [19], Figures 11, 12, 13, 14, 15 and 16. The dolomite $\text{CaMg}(\text{CO}_3)_2$ was present in a smaller percentage and was detected in a single peak $d = (2.88\text{Å})$ at an angle (30.95) 2θ , with an average 4.05%, Table 3, Figures 11 and 16.

3.3.1.2 Quartz

Quartz has the second highest abundance after calcite with an average (53.7%), Table 3. It was found in all selected soil samples except one sample S6, major peak of quartz (4.26Å, 3.34Å, 2.45Å) at an angle of (20.84°, 26.65°, 39.49°) 2θ respectively [20], Figures 11, 12, 13, 14 and 15.

3.3.1.3 Gypsum

Gypsum is a naturally occurring mineral composed of mainly calcium sulfate approximately 79% and water 21%, according to [21]. It is found in all selected soil samples except one samples S1, the average percentage of gypsum is (28.14%), Table 3, Figures 12, 13, 14, 15 and 16.

3.3.1.4 Albite

Albite is a sodium aluminosilicate ($\text{NaAlSi}_3\text{O}_8$), common feldspar mineral, and belongs to the Plagioclase- Feldspar group. Albite was found in three samples (S1, S2, and S3) with an average is (15.1 %), Table 3, Figures 12, 13 and 14.

3.3.1.5 Halite

Halite (NaCl) appears in S16 sample upon basal reflection (2.81Å) at an angle (31.75 2θ). With percentage (16.4%), Table 3, Figure 16.

3.3.1.6 Rutile

Rutile has a chemical composition of TiO_2 and exhibits a vivid red-yellow color, The percentage of Rutile mineral is (2%) in one sample S14, Table 3, Figure 15.

Table 3. Semi-quantitative percentage of clay minerals and non-clay minerals.nh

Sample ID	Calcite	Quartz	Gypsum	Albite	Dolomite	Halite	Rutile	Palygorskite	Chlorite	Illite
S1	7	75.4	-	12.5	1.6	-	-	3.5	-	-
S2	15.7	52.5	2.9	19	-	-	-	3.8	3.1	2.9
S3	25.5	38.3	15.5	14	-	-	-	3.3	3.4	-
S13	15.6	71.3	8.8	-	-	-	-	4.2	-	-
S14	10.4	31	51.9	-	-	-	2	4.7	-	-
S16	9.1	-	61.6	-	6.5	16.4	-	3.8	9.1	-
Average	13.8	53.7	28.14	15.1	4.05	16.4	2	3.8	5.2	2.9

3.3.2 Clay Minerals

Clay minerals were not found in all samples of the study area. The results of XRD technique analysis showed the presence of clay minerals (Palygorskite, Chlorite, Illite), Figures 11, 12, 13, 14, 15 and 16.

3.3.2.1 Palygorskite

The chemical composition of palygorskite is $(\text{Mg, Al})_2\text{Si}_4\text{O}_8(\text{OH})_4\text{H}_2\text{O}$. It is created during the process of weathering of ferromagnesian rocks such as pyroxene and amphibole [22]. Most primary palygorskite deposits were initially created through the conversion of montmorillonite in expansive oceans, either through hydrothermal modification of volcanic substances or as chemical deposits in shallow oceans [23]. The optimal conditions for the in-situ formation of it are a high pH environment rich in silica and magnesium [24]. It is recognized in basal reflection ($d=10.5\text{\AA}$) at an angle ($8.5\ 2\theta^\circ$). the substance remained unaffected when exposed to ethylene glycol. However, the reflection of the base dropped and slightly deviated upon heating to 550°C as a result of water loss [25]. Palygorskite an average is (3.8%), Table 3, Figure 11, 12, 13, 14, 15 and 16.

3.3.2.2 Chlorite

The chlorite group is a variety of similar minerals with a significant chemical variation. Chlorite is formed by weathering of ferromagnesian rocks rich in calcium and iron magnesium at a similar condition to montmorillonite produce but with a higher temperature, alkalinity, and salinity [26]. Chlorite is identified by its basal reflection ($14.7\text{\AA}$) at angle of $2\theta^\circ$ ($6.12\text{\AA}$) which increases considerably when heated over 550°C [27]. Chlorite found in three samples (S2, S3, and S16). The average percentage of chlorite is (5.2%), Table 3, Figures 12, 13 and 16.

3.3.2.3. Illite

Illite is identified readily in basal reflection ($10\text{\AA}$ and $4.9\text{\AA}$) at angle of $2\theta^\circ$ (8.6° , 27.6°) [28]. Illite is a primary clay mineral in mudstone. The Illite group is formed by the weathering of silicate (primarily feldspar) [29]. According to [30], Illite is formed by weathering of the old sedimentary rocks. The optimum conditions for Illite forming are a dry environment rich in potassium ions, high pH (more than 8.5), and low rainfall [31]. Illite is found in one sample S2, Figure 12, with a percentage (2.9 %), Table 3.

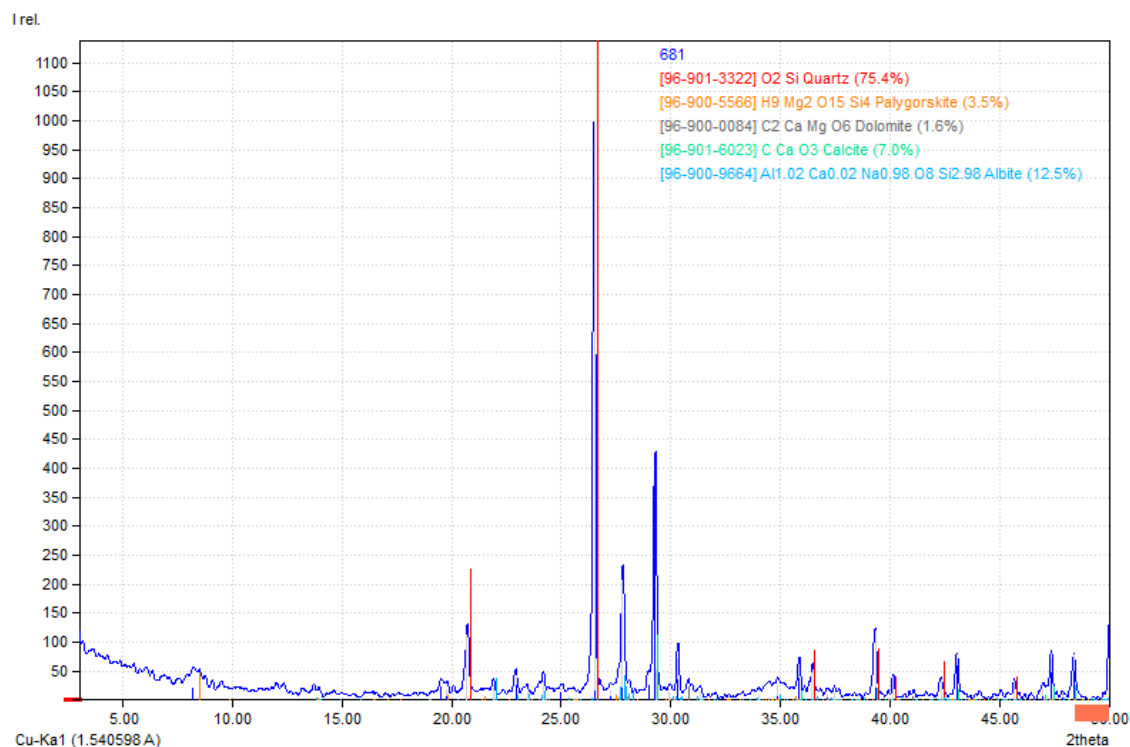


Figure 11. XRD of the bulk sample of S1

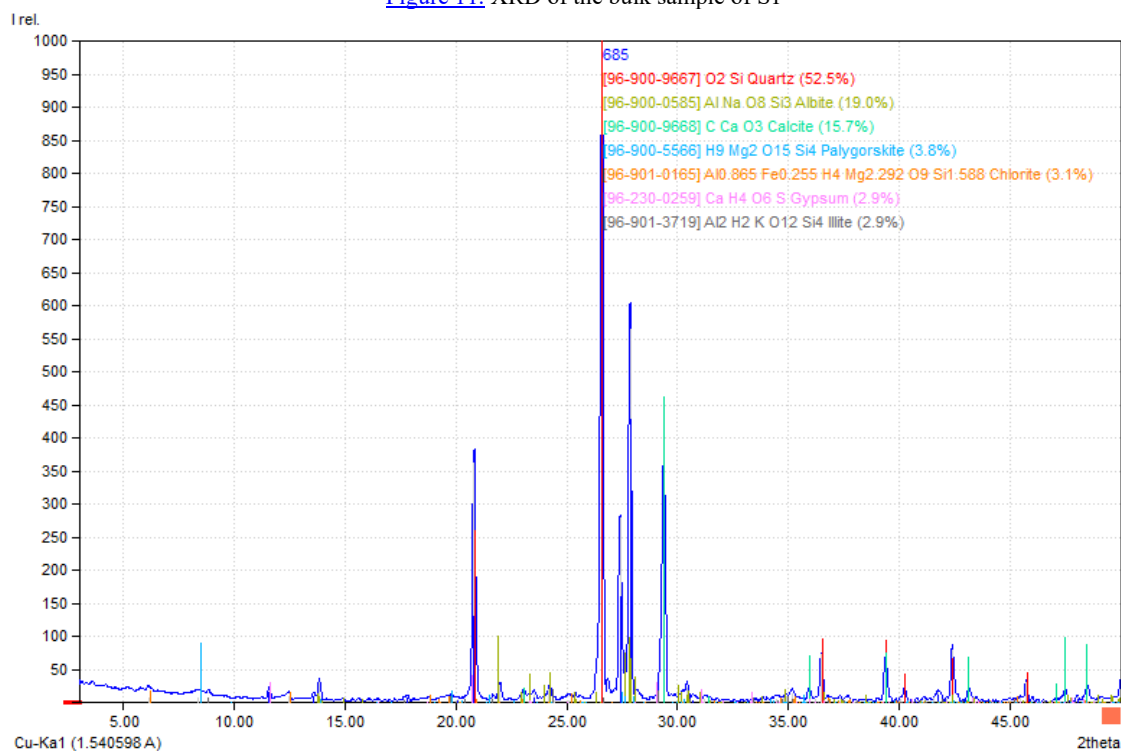


Figure 12. XRD of the bulk sample of S2

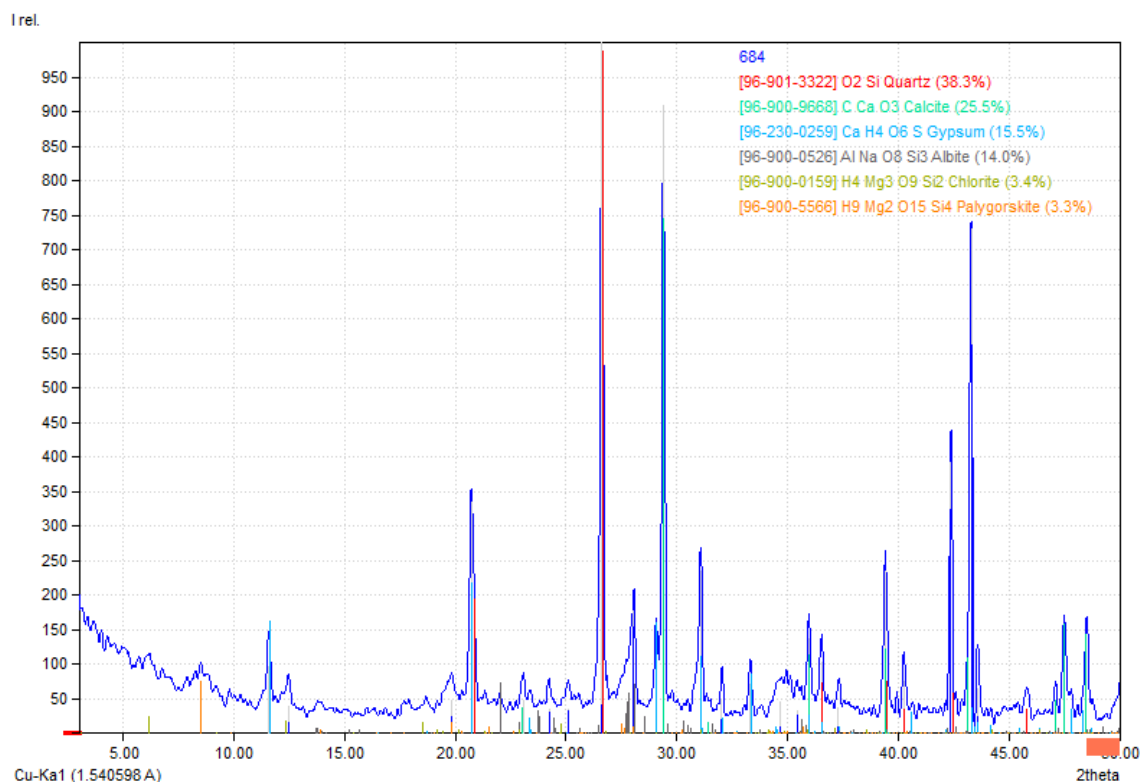


Figure 13. XRD of the bulk sample of S3.

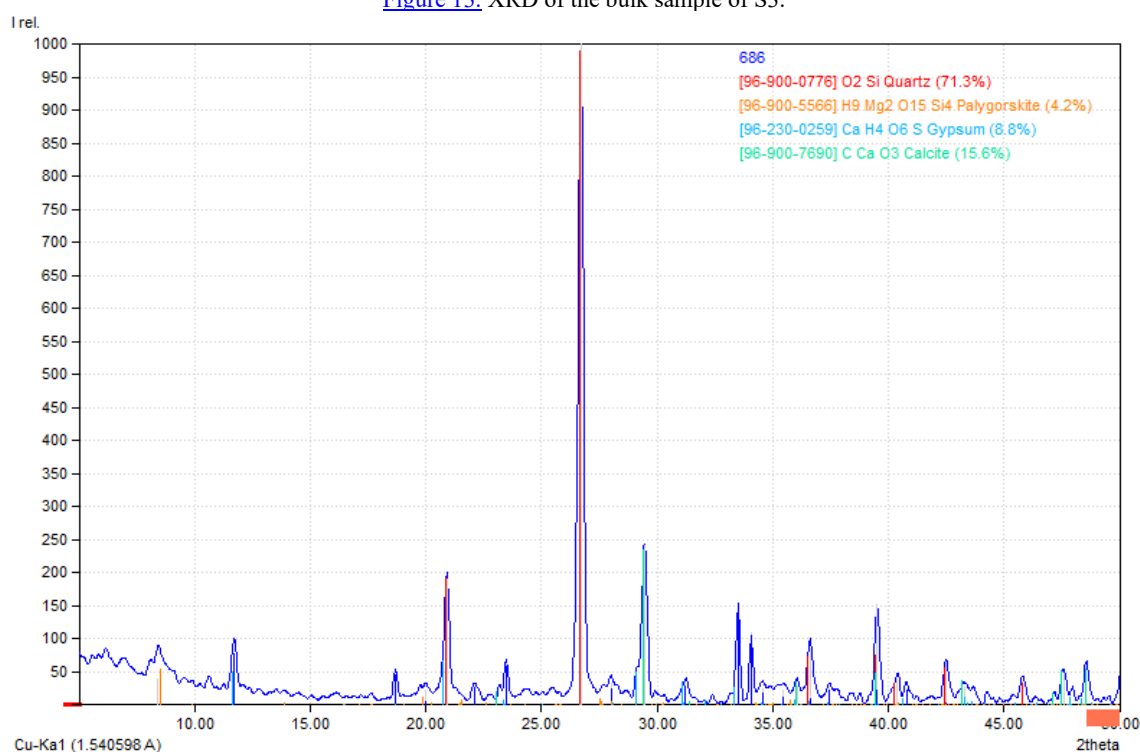


Figure 14. XRD of the bulk sample of S13.

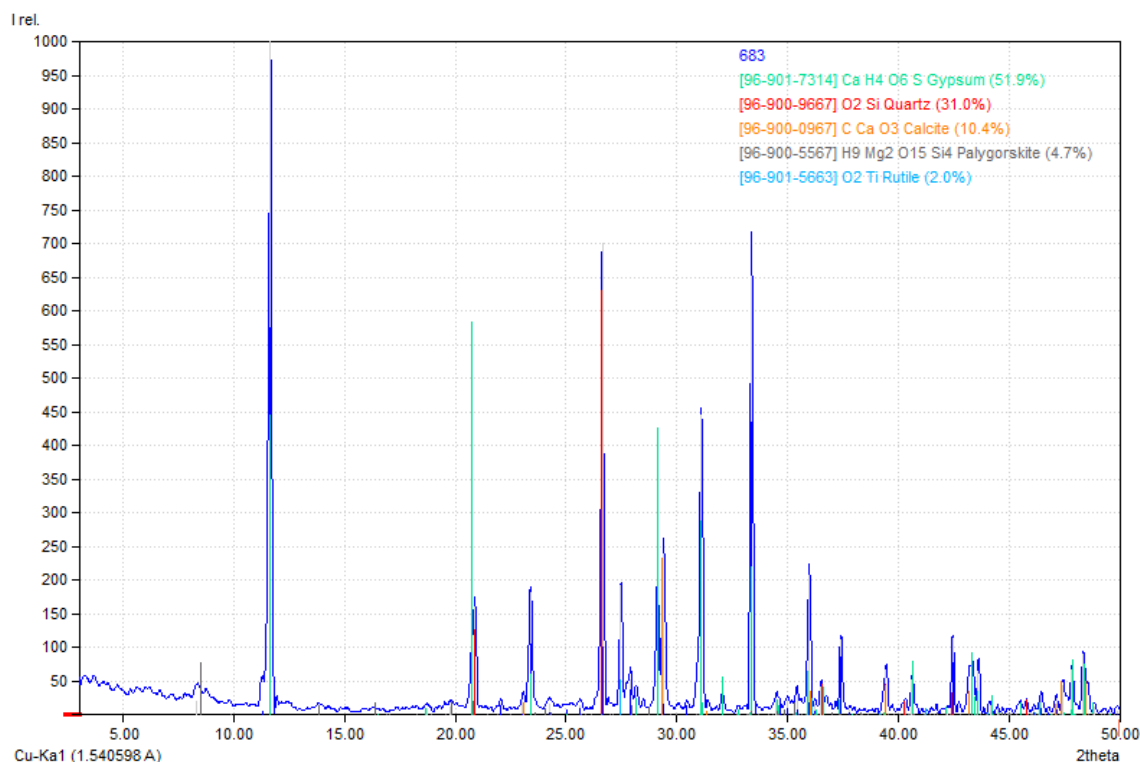


Figure 15. XRD of the bulk sample of S14

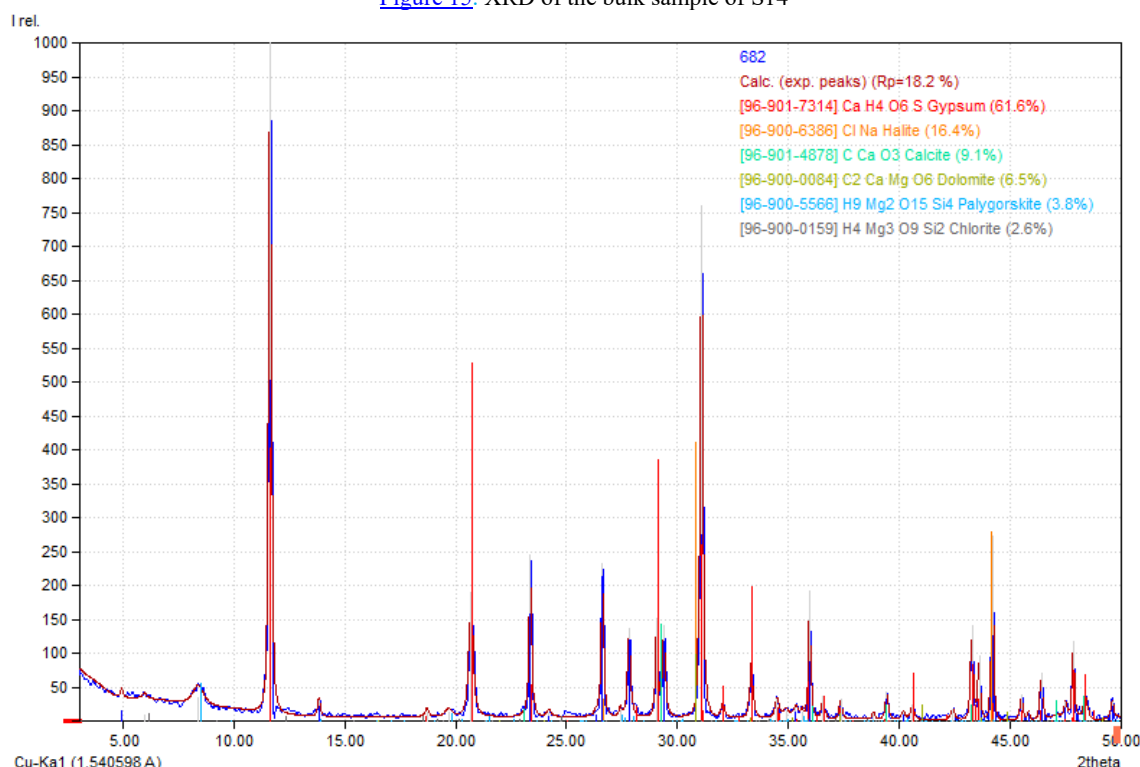


Figure 16. XRD of the bulk sample of S16.

3. CONCLUSION

The results of the geochemical analysis of the major oxides showed a high concentration of SiO_2 , CaO , and a high percentage of loss on ignition compared to other major oxides (Al_2O_3 , SiO_2 , Fe_2O_3 , CaO , MgO , Na_2O , K_2O , SO_3 , and LOI) respectively. Increased the concentration of SiO_2 is due to its existence in the form of free silica (Quartz mineral). and increased CaO indicate to a significant presence of carbonate minerals, specifically calcite and dolomite.

The results of mineralogical analysis revealed that the Quartz, gypsum and calcite minerals were the most abundant non-clay minerals, followed by Albite, Dolomite, Halite, Rutile minerals with lower percentages, and clay minerals are represented by lower percentages of chlorite, Palygorskite and Illite. In this case clay minerals are not considered a controlling factor in the distribution of heavy metals in soils of study area.

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