Alqalam Journal of Medical and Applied Sciences. 2025;8(4):2181-2189
https://doi.org/10.54361/ajmas.258415

Original article

The Detection of the Types and Contents of Metal Oxides and Ores in The Southern Regions of Al-Gabal Alkhder (Libya) Areas

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Abstract
This study was carried out on surface and subsurface samples of soil collected from an area extending between Al–Mkhilly to Tobrouk (Eastern North) of Libya. The main aim of this study is to determine the types and contents of metal oxides in the studied samples by using one of the Nano technologies called (X Ray) Fluorescence (XRF). The results of study showed presence of different types of metal oxides including:(Calcium Oxide, Silicon Oxide Magnesium Oxide, Aluminum Oxide, Ferric Oxide, Manganese Oxide, Titanium Oxide, Potassium Oxide, Phosphorus Oxide and Sulfur Oxide) (CaO, SiO_2, MgO, Al_2O_3, Fe_2O_3, MnO, TiO_2, K_2O, P_2O_5, SO_3) beside important contents of $CaCO_3$and Mg CO_3. The contents of higher detected oxides as (SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, $CaCO_3$ and Mg CO_3.of the surface samples were ranged as following (14.22 -67.15 %), (2.5 -9.17 %), 1.27 -4.39 %), 11.04 -40.86 %), (1.73 -2.70 %), (19.3 -72.58 %) and (3.60 -5.64 %), respectively. On the other side, their contents in subsurface samples were fluctuated in the ranges of (4.5 -66.95 %), (1.35 -9.92 %), (0.61 -4.59 %),12.99 -48.96 %), (0.64 -2.83 %), (19.83 -87.05 %), and (1.33 -5.92 %), respectively. Generally, the results of this study showed different values of metal oxides, the main metal Ores may include. Calcite / or Dolomite, Hematite, Quartz, Alumina, Feldspar, these oxides are very important in the industries.
Keywords. Metal Oxides, Ores, XRF, Southern, AlGabal Alkhder, Libya.

Introduction

Oxides are the most abundant materials in the Earth's crust and represent a broad group of relatively stable compounds formed through the chemical union of oxygen with another element or an organic compound. Water serves as an example of an inorganic oxide, being hydrogen oxide, as does carbon monoxide. Theoretically, all elements can combine with oxygen to yield oxides. Hydrogen may also combine with oxygen in reactions to form hydroxides or oxy hydroxides, exemplified by aluminum hydroxide Al $(OH)_3$ and aluminum oxy hydroxide AlOOH, respectively; these can be grouped under the general term oxides. Furthermore, oxides exhibit high chemical stability in many environmental systems, such as the Earth's atmosphere, which explains their pronounced chemical stability. Chemically, oxides comprise at least one type of metal and oxygen. However, numerous mixed metal oxides can also exist, for example, $FeTiO_3$. Oxides can also be classified according to the bonding types between the cation and oxygen into ionic oxides and covalent metal oxides [1].

Silica (silicon oxide), SiO_2, occupies a special position among metal oxides in general, being the most abundant. It occurs in diverse natural mineral forms and can be found in nature either in an amorphous state or in crystalline forms (for example, such as quartz, cristobalite, and tridymite), with crystalline silica, particularly quartz, being the most common. It is a common compound in soils, sands, and rocks, whereas amorphous silica is commonly deposited by living organisms, including plants, unicellular organisms such as diatoms, and multicellular organisms like sponges. SiO_2 also features a significant "reverse" pathway, from living organisms to the "non-living" nature: for example, diatomite rock is in fact a sedimentary rock composed primarily of the skeletal remains of diatoms [2]. Metal oxides are among the most important and distinctive solid catalysts. Metal oxides act as heterogeneous catalysts and are used for acid-base reactions and oxidation-reduction reactions.

Certain groups of metals, especially transition metals, have attracted considerable interest due to their outer electronic configuration. They are widely applied in various reactions, including oxidation, dehydrogenation, and oxide formation. Transition metal oxides such as tungsten oxide and titanium oxide, WO_3, TiO_2, are widely used as heterogeneous nanoscale acid catalysts [3]. Mesoporous metal oxides are renowned for their special structural properties, such as high surface area, variable pore size, and stability. It has been demonstrated that an association exists between Mn (III) and Mn (IV) with hematite ore (one of the iron ores) to produce effective oxidation [4].

Many elements occur as oxides in various types of rocks and salts, such as nickel oxides, a mineral widely used in mineralogy. It is also a micronutrient for plants, although it is toxic to them at high concentrations. Zinc and its oxides are essential minerals for both plants and animals, but can be harmful to both at high concentrations. The importance of some oxides varies compared to others; for example, manganese oxides decrease in importance when compared to iron or aluminum oxides in the adsorption process, while gibbsite (aluminum hydroxide) ranks second in importance. Oxides of metallic elements

Received*: 02-08-2025 -* ***Accepted****: 01-10-2025 -* ***Published****: 09-10-2025*

https://doi.org/10.54361/ajmas.258415

often play a significant role in ores, many of which occur either as discrete oxides or as mixed oxides with each other, which imparts importance to many of them in various industrial applications [5]. The contents of metals and metal oxides were measured in many studies on some Libyan samples [6-36], using different methods as atomic absorption spectroscopy (AAS), ion-coupled plasma (ICP), X-Ray Fluorescence (XRF) for determining metals and metals oxides were applied on different samples, also radioactive elements were detected in different samples. In studies of natural or chemical compounds were conducted, including many studies about different compounds as natural radionuclides [37-40], Hydrocarbons [41-49], plants [50-81 and others [82-95]. The objectives of the study are summarized in the following points: To determine the concentrations of natural oxides in the study area by identifying their components quantitatively and qualitatively using X-ray Fluorescence (XRF), one of the nanotechnology techniques. And to clarify the most important mineral ores present in the study area by identifying the types of most abundant oxides, which constitute the primary ores in the samples.

Study area description

This study was conducted in the region extending from Al-Mukhaili to Tobruk in eastern Libya. Ten sampling sites were established, with both surface and subsurface samples collected from each location. The distance between consecutive sites was 15 km, covering a total study area length of approximately 150 km (Figure 1).

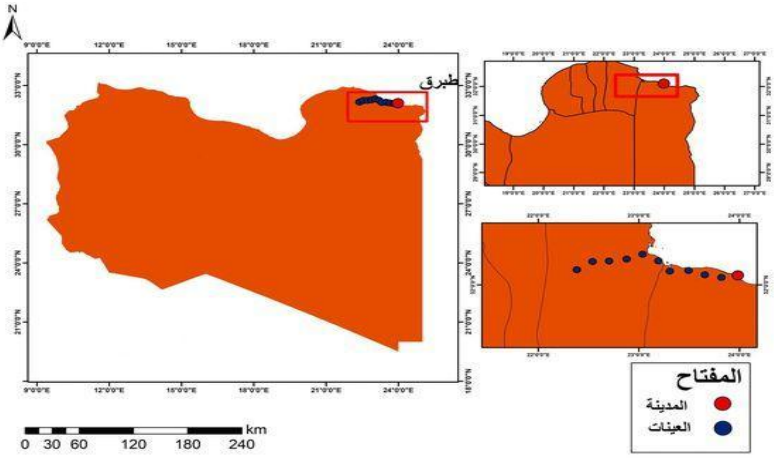

Figure 1. The map of Study Locations

Sample Collection Methods

The study area was divided into ten sites. Soil samples were collected from each site at two different depths: a surface sample (0–10 cm) and a subsurface sample (15–20 cm). Each sample was placed in a tightly sealed plastic container, and all relevant data, including sample number, distance, and depth classification (surface/subsurface), were recorded. The study sites were coded and labeled as follows:
Site (1): Al-Mukhaili, Site (2): Al-Azzayat, Site (3): Martubah, Site (4): Umm Razm, Site (5): Bomba Bay, Site (6): Al-Tamimi, Site (7): Ain Ghazalah, Site (8): Balkhather, Site (9): Al-Mursas, Site (10): Tobruk. Samples were subsequently transported to the chemical laboratory of the Al-Fataih Cement Factory in Derna for analysis. The analyses included quantifying the following element oxides: SiO_2, MgO, CaO, Fe_2O_3, Al_2O_3, $CaCO_3$, $MgCO_3$, P_2O_5, MnO, Al_2O_3, TiO_2, SO_3, K_2O.

Analytical Methods

Approximately 10 g of soil sample was placed in a vibrating mill device for thorough grinding prior to pressing. The ground sample was then transferred to a press device and compacted into a small, cohesive disk. The sample disk was placed in an X-ray device (Burker X-Ray), and readings for the aforementioned oxide concentrations were recorded [8].

Results

The results for oxide concentrations, categorized by sample type (surface/subsurface soil), are as follows (Tables 1&2):

Table 1. The contents of SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO and K_2O

Type Metal Oxide	Surface samples %	Subsurface samples %
SiO_2	14.22 -67.50	4.75 – 66.95
Al_2O_3	2.5 -9.17	1.35 -9.92
Fe_2O_3	1.27 – 4.39	0.61 -4.54
CaO	11.04 - 40.86	12.99 – 48.96
MgO	1.73 – 2.70	0.64 – 2.83

***Received**: 02-08-2025 - **Accepted**: 01-10-2025 - **Published**: 09-10-2025*

https://doi.org/10.54361/ajmas.258415

Table 2. The contents of SO_3, TiO_2, P_2O_5, $CaCO_3$ and $MgCO_3$

Type Metal Oxide /CO_3	Surface samples %	Subsurface samples %
SO_3	0.18 -0.287	0.17 -0.42
TiO_2	0.20 -0.75	0.07 -0.69
P_2O_5	0.087 -0.147	0.06- 0.150
$CaCO_3$	19.37 – 72.58	19.83 – 87.05
$MgCO_3$	3.60 -5.64	1.33 - 5.92

Silicon Oxide (SiO_2) Analysis

There were differences observed between sites for SiO_2, detected in all surface and subsurface samples (Table 1). Concentrations ranged from 14.22% to 67.15% (surface) and 4.75% to 66.95% (subsurface). Highest concentration: 67.1% at Site (10) Tobruk (surface). Lowest concentration: 14.22% at Site (2) Al-Mukhaili (surface). Subsurface concentrations peaked at 66.95% (Site 10), with the lowest value at Site (1). Relatively elevated SiO_2 levels occurred at Sites (8) and (9) (Ain Ghazalah and Al-Mursas), near Tobruk. A general west-to-east increasing trend in SiO_2 concentrations was observed across the study area.

SiO_2 ranked second in abundance among oxides in the study area. Its presence is attributed to its status as a primary constituent of Earth's crust. Recorded values align with studies reporting high SiO_2 concentrations in soils (Branowski et al., 2002). Distribution patterns correlate with soil type and study location, consistent with findings from Turkish regions where SiO_2 presence depends on local mineral deposits [96]. Positive correlations were identified between SiO_2 and Al_2O_3/MnO. Negative correlation was observed between SiO_2 and calcium carbonate ($CaCO_3$), indicating that higher SiO_2 concentrations correspond to lower $CaCO_3$ levels.

The obtained results for aluminum oxide revealed its presence in all surface and subsurface samples, with concentrations ranging between (2.5% - 9.17%) and (1.35% – 9.92%), respectively. The highest values (concentrations) were recorded at Site (10) Tobruk, measuring (9.17%), while the lowest concentrations occurred at Site (7) in surface samples (the area south of Martubah). Subsurface concentrations peaked at (9.92%) at Site (10), with the minimum value at Site (1) (Al-Mukhaili region). Overall, the results indicated highly significant variations in aluminum oxide concentrations across the study area. When comparing aluminum oxide (Al_2O_3) concentrations in the study regions, it ranked fifth in abundance. The presence of aluminum oxides in soil samples is generally attributed to their status as primary constituents of Earth's crust. The values recorded in this study align with previous research demonstrating elevated aluminum oxide concentrations in soil samples, and their distribution depends on soil type and study location across global regions [97].

Statistical analyses further indicated a positive correlation between aluminum oxide content and iron and manganese oxides, suggesting co-enrichment of these oxides within the study area.

The obtained results for iron oxide revealed its presence in all surface and subsurface samples, with concentrations ranging between (1.27% - 4.39%) and (0.61% – 4.59%), respectively. The highest values (concentrations) were recorded at Site (10) Tobruk, measuring (4.39%), while the lowest concentrations occurred at Site (7) in surface samples. Subsurface concentrations peaked at 4.59% at Site (8) (Al-Mursas region), with the minimum value at Site (1) (Al-Mukhaili). Overall, the study results indicated highly significant variations in iron oxide concentrations across the study area ($p < 0.01$). When comparing iron oxide (Fe_2O_3) concentrations in the study regions, it ranked sixth in abundance. The presence of iron oxides in soil samples is generally attributed to their status as primary constituents of Earth's crust. The values recorded in this study align with previous research demonstrating elevated iron oxide concentrations in soil samples, and their distribution depends on soil type and study location across global regions. Statistical analyses further indicated a strong correlation between iron oxide content and other oxides: manganese oxide ($r = 0.85$) and potassium oxide ($r = 0.95$), and iron oxides and manganese oxides often co-occur.

The obtained results for calcium oxide revealed highly significant differences in all surface and subsurface samples, with concentrations ranging between (11.04% - 40.86%) and (12.99% – 48.96%), respectively. The highest values (concentrations) were recorded at Site (7) (Ain Ghazalah), measuring (40.86%), while the lowest concentrations occurred at Site (10), Tobruk, in surface samples. Subsurface concentrations peaked at (48.96%) at Site (1) (Al-Mukhaili), with the minimum value at Site (10) (Tobruk). The study results recorded reduced concentrations at Sites (8 and 9) (Balkhather and Al-Mursas regions). Calcium concentrations were comparable across the remaining study areas (south of Al-Azzayat, Martubah, Umm Razm, and Al-Tamimi). When comparing calcium oxide (CaO) concentrations in the study regions, it ranked third in abundance. The presence of calcium oxides in soil samples is generally attributed to their status as primary constituents of Earth's crust. The values recorded in this study align with prior research demonstrating elevated calcium oxide concentrations in soil samples, with distribution patterns dependent on soil type and geographic location across global regions [96]. Statistical analyses indicated a strong positive correlation between calcium oxide content and calcium carbonate, likely due to the occurrence of

***Received**: 02-08-2025 - **Accepted**: 01-10-2025 - **Published**: 09-10-2025*

calcium in the form of calcite ore ($CaCO_3$) associated with calcium oxide in the study area samples. The results indicated highly significant differences in magnesium oxide across all surface and subsurface samples and study regions, with concentrations ranging between (1.73% - 2.70%) and (0.64% – 2.83%), respectively. The highest values (concentrations) were recorded at Sites (3–8) in the study areas (Martubah, Umm Razm, Al-Tamimi, Bomba Bay, and Ain Ghazalah), measuring (2.70%), while the lowest concentrations occurred at Site (1), Al-Mukhaili, in surface samples. Subsurface concentrations peaked at (2.83%) with the minimum value at Site (1), and to a lesser extent at Al-Mursas, Site (9). When comparing magnesium oxide (MgO) concentrations in the study regions, it ranked seventh in abundance. The presence of magnesium oxides in soil samples is generally attributed to their status as primary constituents of Earth's crust. The values recorded in this study align with prior research demonstrating elevated magnesium oxide concentrations in soil samples. Statistical analyses indicated a positive correlation between magnesium oxide content and other oxides (K_2O, TiO_2, MnO).

The obtained results revealed statistically significant differences in potassium oxide concentrations across all surface and subsurface samples, with concentrations ranging between (0.47% - 1.65%) and (0.49% – 2.06%), respectively. The highest values (concentrations) were recorded at Site (3) (Martubah), measuring (2.06%), and in the Umm Razm region for surface samples (1.65%), while the lowest concentrations occurred at Site (7) (Ain Ghazalah) in surface samples. Subsurface concentrations were generally higher than surface concentrations across most study sites. Overall, the study results did not indicate significant variations in potassium oxide content across the study area. When comparing potassium oxide (K_2O) concentrations in the study regions, it ranked eighth in abundance. The presence of potassium oxides in soil samples is generally attributed to their status as primary constituents of Earth's crust. The values recorded in this study align with prior research demonstrating low potassium oxide concentrations in soil samples where present [96]. Statistical analyses indicated a strong positive correlation between potassium oxide content and other oxides (K_2O, TiO_2, MnO), while concurrently revealing a negative correlation between potassium oxide (K_2O) and calcium carbonate ($CaCO_3$), suggesting that higher K_2O concentrations correspond to lower $CaCO_3$ levels in the study area.

The obtained results for sulfur oxide revealed its presence in all surface and subsurface samples, with concentrations ranging between (0.18% - 0.287%) and (0.173% – 0.427%), respectively. The highest values (concentrations) were recorded at Site (1) in the Al-Mukhaili region, measuring (0.427%) in surface samples, while subsurface concentrations peaked at (0.173%). Overall, sulfur oxide concentrations were comparable across most study sites. The study results did not indicate significant variations. When comparing sulfur oxide (SO_3) concentrations in the study regions, it ranked eleventh in abundance. The values recorded in this study align with previous research demonstrating elevated sulfur oxide concentrations in soil samples [97]. Statistical analyses indicated a strong positive correlation between sulfur oxide content and other oxides (K_2O, TiO_2, MnO), while concurrently revealing a negative correlation between sulfur oxide (SO_3) and calcium carbonate ($CaCO_3$), suggesting that higher SO_3 concentrations correspond to lower $CaCO_3$ levels in the study area.

The obtained results for titanium oxide revealed its presence in all surface and subsurface samples, with concentrations ranging between (0.20% - 0.75%) and (0.07% – 0.69%), respectively. The highest values (concentrations) were recorded at Site (10) Tobruk, measuring (0.75%), while the lowest concentrations occurred at Site (7) (Ain Ghazalah) in surface samples. Subsurface concentrations peaked at (0.69%) with the minimum value at Site (1) (Al-Mukhaili). Overall, the study results did not indicate significant variations in titanium oxide concentrations across the study sites, except for its relative elevation at Site (10) and Site (8) (Balkhather region. The obtained results for manganese oxide revealed its presence in all surface and subsurface samples, with concentrations ranging between (0.025% - 0.065%) and (0.014% – 0.075%), respectively. The highest values (concentrations) were recorded at Sites (3 and 10) in the Martubah and Tobruk regions, measuring (0.065%), while the lowest concentrations occurred at Site (7) in surface samples. Subsurface concentrations peaked at (0.075%) with the minimum value at Site (1). Manganese oxide concentrations were comparable across the remaining study sites, and no significant variations were recorded. When comparing manganese oxide (MnO) concentrations in the study regions, it ranked thirteenth in abundance. The presence of manganese oxides in soil samples is commonly associated with iron oxide. Statistical analyses indicated a strong positive correlation between manganese oxide content and other oxides, particularly iron oxide ($r = 0.98$), suggesting that manganese oxide occurrence is more pronounced in areas with elevated iron oxide concentrations within the study region.

The obtained results for phosphorus oxide revealed its presence in all surface and subsurface samples, with concentrations ranging between (0.087% - 0.147%) and (0.067% – 0.157%), respectively. The highest values (concentrations) were recorded at Site (7) (Ain Ghazalah), measuring (0.147%), while the lowest concentrations occurred at Site (10), Tobruk, in surface samples. Subsurface concentrations peaked at (0.157%) with the minimum value at Site (10) in the Tobruk region. The study results did not indicate significant variations in phosphorus oxide concentrations across the study area. When comparing phosphorus oxide (P_2O_5) concentrations in the study regions, it ranked twelfth in abundance. The presence of phosphorus oxides in soil samples is generally attributed to their status as primary

***Received**: 02-08-2025 - **Accepted**: 01-10-2025 - **Published**: 09-10-2025*

https://doi.org/10.54361/ajmas.258415

constituents of Earth's crust, occurring in variable quantities according to the distribution of their ores within the study area. Statistical analyses indicated a positive correlation between phosphorus oxide content and other oxides.

The obtained results for calcium carbonate revealed its abundant presence in all surface and subsurface samples, with concentrations ranging between (19.37% - 72.58%) and (19.83% – 87.05%), respectively. The highest values (concentrations) were recorded at Site (1) (Al-Mukhaili), measuring (87.05%), while the lowest concentrations occurred at Site (10) (Tobruk) in surface samples. Subsurface concentrations were higher compared to surface samples, with the minimum value at Site (10). The study results indicated highly significant variations in calcium carbonate concentrations across the study area. When comparing calcium carbonate ($CaCO_3$) concentrations with other oxides in the study area, it ranked first in abundance. The values recorded in this study align with prior research demonstrating elevated calcium carbonate levels in soil samples [95], and its distribution depends on soil type and geographic location across global regions. Statistical analyses indicated a strong positive correlation between calcium carbonate content and other oxides: K_2O, TiO_2, and MnO.

The obtained results for magnesium carbonate revealed its presence in all surface and subsurface samples, with concentrations ranging between (3.60% - 5.64%) and (1.33% – 5.92%), respectively. The highest values (concentrations) were recorded at Site (4) (Umm Razm), measuring (5.64%), while the lowest concentrations occurred at Site (1) Al-Mukhaili in surface samples. Subsurface concentrations peaked at (5.92%) with the minimum value at Site (1) in the Al-Mukhaili region. The study results did not indicate significant variations in magnesium carbonate concentrations across the study area. When comparing magnesium carbonate ($MgCO_3$) concentrations in the study regions, it ranked second in abundance. The presence of magnesium carbonate in soil samples is generally attributed to its status as a primary constituent of Earth's crust, occurring in variable quantities according to the distribution of its ores within the study area. Statistical analyses indicated a positive correlation between magnesium carbonate content and other oxides.

Discussion

This study revealed high concentrations of calcium carbonate, silicon oxide, and calcium oxide, with lower levels of magnesium oxide, sulfur, and magnesium carbonate across most study sites. The distribution of each oxide concentration at every site demonstrates the dominance of calcium carbonate and calcium oxide in the study area. Results further indicated elevated levels of silicon oxide, aluminum oxide, and iron oxide in the study site samples, alongside notable magnesium carbonate concentrations. Conversely, concentrations of other elements, including sodium, potassium, manganese, and titanium oxides, were relatively low. Soil types commonly comprise mixtures of elements formed through geological and chemical processes. Numerous factors, including physical and chemical properties and microorganisms, influence elemental distribution in soils [96].

Some studies highlight that soil's ecological and geological systems constitute an interconnected, complex framework akin to aquatic systems, due to interactions such as ion exchange between cations and anions, rock types and their mineral content, pH levels, and various physicochemical and biological interferences. However, soils differ from aquatic and atmospheric systems due to additional influencing factors like climate change, which impacts chemical reactions governing elemental distribution. Modern analytical techniques for soil components, such as X-ray fluorescence (XRF), X-ray diffraction (XRD), and inductively coupled plasma (ICP) analysis, have enabled more precise characterization of soil and rock compositions than traditional methods [97]. Based on the recorded results, elevated concentrations of silicon oxide (SiO_2), calcium oxide (CaO), and calcium carbonate ($CaCO_3$) are primarily attributed to the presence of limestone or calcareous sandstone ores [98]. Trace concentrations of elements like TiO_2 alongside Fe_2O_3 likely stem from their incorporation into clay mineral structures within the study area. Additional concentrations of K_2O or Na_2O are often linked to granite rocks containing these elements [8].

The ubiquitous presence of calcium oxide (CaO) and magnesium oxide (MgO) in all samples is typically associated with calcite ($CaCO_3$) or dolomite [$CaMg(CO_3)_2$] ores, carbonate weathering, and ion exchange with other ions. Some studies suggest that reduced aluminum oxide levels in certain soils may result from its accumulation in specific plant species. Silicon and aluminum generally originate from aluminosilicate ores or as carbonates of the aforementioned minerals. Weathering of silicon or aluminum carbonate salts often generates elemental oxides, including Al_2O_3 and SiO_2, which may accumulate in plants across some study areas. The solubility of certain elements depends on factors like pH, leading to concentration variations between regions. Low K_2O concentrations typically result from ion exchange processes when calcium or magnesium levels are high, coupled with potassium accumulation in plants, causing marked depletion relative to other elements.

Iron oxide concentrations generally correlate with common iron ores like magnetite (Fe_2O_3). Some studies indicate interactions between elements and soil organic matter, facilitating their presence in various chemical forms, including oxides. The presence of manganese oxides in most samples is primarily attributed to its unique chemical structure, existing in multiple oxidation states (Mn^{+7}, Mn^{+5}, Mn^{+3}, Mn^{+2}).

***Received**: 02-08-2025 - **Accepted**: 01-10-2025 - **Published**: 09-10-2025*

This characteristic enables the formation of diverse oxides with oxygen in various soil types. Additionally, the occurrence of elemental ores within the soil samples, combined with the soil's inherent nature, whether sandy, clayey, or rocky, significantly influences the distribution of minerals, their oxides, or hydroxides. Elemental distribution in soils is further affected by spatial characteristics and specific plant species [20].
The relative distribution of the most abundant oxides and compounds in the study area: calcium carbonate, silicon oxide, ferric oxide, calcium oxide, aluminum oxide, and magnesium carbonate. These figures confirm that calcium carbonate and calcium oxide concentrations were elevated across all study sites. These compounds are primary materials in cement manufacturing, and calcium oxide ores (limestone) serve as vital resources for construction materials. The high silica (quartz) concentrations, particularly in the eastern study sites (Sites 9–10: Al-Mursas and Tobruk), represent a significant resource for industries such as glass manufacturing. The study also identified notable concentrations of aluminum oxide and ferric oxide, which hold importance in numerous industrial applications. The occurrence of mineral ores in the study area presents opportunities for economic exploitation either through direct industrial use or element extraction and export, positioning these resources as critical mineral wealth.

Conclusion

This study, conducted to determine concentrations and types of elemental oxides in the study area, demonstrates that the employed method, X-ray fluorescence (XRF) analysis, yielded reliable and sensitive results. It confirmed the presence of diverse elemental oxides, many of which hold significance in strategic and other key industries. The study further identified abundant mineral ores in the study region, including calcite, limestone, quartz, hematite, gibbsite, and others. These ores can be utilized in various industries, such as glass, cement, and metallic conductor manufacturing. Given the importance of accurately quantifying natural components in systems like soils and rocks, the study's results support the use of advanced analytical techniques to identify rare elements that represent vital energy, economic, or industrial resources. Recommended methodologies include: Inductively Coupled Plasma (ICP) analysis for precise elemental quantification. Scanning Electron Microscopy (SEM), a nanotechnology technique, is used to further elucidate the chemical and geological structures of Libyan soils and rocks.

Acknowledgments

The participating authors in this study express their gratitude and appreciation to the staff of the chemical laboratory at the Derna Cement Factory for their cooperation during the completion of this research.

Conflicts

No conflicts of interest exist regarding the results of this study.

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Received*: 02-08-2025 -* ***Accepted****: 01-10-2025 -* ***Published****: 09-10-2025*

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***Received**: 02-08-2025 - **Accepted**: 01-10-2025 - **Published**: 09-10-2025*