Alqalam Journal of Medical and Applied Sciences. 2025;8(3):1316-1321
https://doi.org/10.54361/ajmas.258309

Original article

Contents of Metal Oxides in Marine Sediment and Rock Samples from the Eastern Libyan Coast, Utilizing the X-ray Method

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Abstract
In this study, the contents of metal oxides and chemical composition of marine sediments were studied in some sediment samples collected from the Eastern coast near Tobrouk region (Libya). Different samples were collected during the summer of 2024. The X-ray method of analysis was used to estimate the types of metal oxides and heavy metal oxides in the studied samples. The results recorded that the samples containing different forms of metal oxides including: (NaO, MgO, Al_2O_3, SiO_2, SO_3, CaO, $CaCO_3$, Fe_2O_3, CuO, and ZnO) and their presents were in percentage as follow: (0.2, 0.7, 1.6, 0.2, 0.6, 11.1, 83.0, 0.1,0.3 and 0.2 %) for the oxides, respectively. On the other side, the same elements oxides were recorded for the Rocks sample, the concentrations of the Metal oxides are: (0.1, 2.3, 1.2, 2.7, 0.6, 10.5, 79.7, 0.5, 0.6, and 0.4%). The findings demonstrated that rocks and sediment samples had substantial concentrations of calcium carbonate ($CaCO_3$) and metal oxides. However, compared to carbonate, the metal oxides (NaO, MgO, Al2O3, SiO2, SO3, CaO, CuO, ZnO, and Fe2O3) in the sediments and rocks of the samples that were recorded had a lower rate of occurrence. Distribution of oxides in both rock and sediments follows the same trend as follows: $CaCO_3$ >> CaO >>MgO >Al_2O_3 > SO_3. This pattern was found to be the same in both x-ray and chemical analysis, indicating significantly high concentrations of Calcium and Carbonates. The presence of some heavy metal oxides is mainly due to the presence of some pollutants as ZnO, CuO, and FeO.

Keywords. Metal Oxides, Marine Sediments, Libyan, X-Ray.

Introduction

There are different sources of marine sediments, including insoluble materials, marine rocks and soils. Sediments have a high layer value, more than a kilometer thick make up the majority of continental shelves. These sediments come from a variety of sources: Clastic sediments are the final byproducts of weathering that are formed from rock (clast = fragment). Terrigenous elements eroded from land make up the majority of clastic deposits. Clastic sediments are also produced by the erosion of undersea rock and rocky coastlines. Clastic sediments make up to 85% of all coastal marine sediments [1].

Although wind, ice, and waves also play a role, rivers are the primary means of transporting the byproducts of terrestrial rock weathering. Rock pieces continue to break down as a result of the carried particles and fragments abrading one another and eroding the surfaces they pass over. Important variables include time and distance: the longer the trip to the sea, the greater the likelihood that mineral grains will be rounded and smaller due to abrasion. Grain is sorted based on size, shape, and density during this trip, and minerals that are less stable chemically are dissolved. Rock fragments, quartz, and clay minerals are the most frequent solid byproducts of weathering. Quartz is the only communal mineral found in igneous and metamorphic rocks that is both chemically stable and hard (abrasion-resistant). Due to these characteristics, in modern beach and river sand and sediment alongside prehistoric sandstones, quartz is the most common mineral [2]. The atmosphere deposits a little amount of terrigenous material. It is made up of wind-borne dust and volcanic ash, and it mostly consists of very small quartz sand grains, along with soil particles that are abundant in calcium (Ca), iron (Fe), magnesium (Mg), and aluminum (Al)[3].

Except for large deserts like the Sahara, the source of airborne dust may not be of significant influence in the majority of places in our current time, but in the past, it was, and this has caused in the buildup of large amounts of loess, as in the Bohai Sea, China. As erosion of loess deposits, which are ultimately released and causes an abnormally high sediment burden. The flow of meltwater through subglacial tunnels and the height of mountains, as well as snow flow glaciers, produce large volumes of sediment in arctic and subarctic regions. Grain size is a measure of how far along the weathering process is; small-scale grains are the result of the weathering of bigger grains. Clastic sediments can be categorized based on grain size.

Grains of various sizes are typically seen in sediment samples. The largest existing grains (clasts) are used to name sedimentary deposits. The following are typical rock types [3]: including different types of rocks. Large angular pieces make up Breccia, whereas little angular ones make up Gritstone; rounder elements make up Conglomerate. Diamict: unsorted (glacial) sediment that has been indurated. Sandstone is a type of sedimentary rock made up of quartz particles that are the size of sand and finer-grained materials like feldspar, micas, lime, and clay that fill in the spaces between. The minerals that precipitate in the pores cause the cement to solidify. Arkose is a sandstone that contains a lot of feldspar.

Received*: 02-05-2025 -* ***Accepted****: 28-06-2025 -* ***Published****: 05-07-2025*

The biogenic sediments referred to as "bioclastic sediments" because they are made up of pieces of organic skeletons. They are primarily composed of aragonite or calcite crystals, which are forms of calcium carbonate (CaCO3). Other biogenic sediments are referred to as organic-rich biological sediments because they contain a significant amount of organic matter, such as dead plant remains (peat). In coastal areas with limited terrigenous material supply (no nearby river deltas), biogenic sediments may make up a significant portion of the bed material [4].

The manner and source of water bed feeding have a great impact on the components and characteristics of sediment. For example, beaches in tropical and subtropical areas are covered mainly by carbonated sands, which are a product of close reefs that are home to corals, shells, and skeletal debris, and calcium carbonate precipitate [5]. The most common chemical composition of sediment, including calcite ($CaCO_3$), gypsum ($CaSO_4.2H_2O$), and halite (NaCl), is the most commonly produced chemical in this manner. When the water gets saturated, which is frequently the result of evaporation, minerals precipitate out of solution to produce chemical deposits. Calcite ($CaCO_3$), gypsum ($CaSO_4.2H_2O$), and halite (NaCl) are the most prevalent chemical deposits that are created in this manner. They are a typical kind of silt found in back-barrier coastal plains, or sabkhas, that are predominantly available in dry regions.

Sheets of oxides and hydroxides and silica (Si) tetrahedra serve as their fundamental building blocks. Igneous rocks and feldspars weather to produce micas and clay minerals [6]. A brief introduction is given to a number of the physicochemical characteristics of marine. Other coastal provides more thorough explanations. Fall speed: Sediment particles settle out of suspension to form the seafloor of oceans and coastal seas. The primary determinants of which particles settle where are turbulence, availability, and particle fall velocity. Only coarse grades can settle in active conditions. The size and density of silt particles determine their fall velocity. Grain size is crucial since the densities of various sediment types are comparable [6].

The factors that impact sediment transport are strongly connected to the strength of flow close to the bed, and slightly above these contributors are not strongly understood; therefore, modules are used to simplify and describe sediment transport [7-9]. The settling of sediment on the sea bed and coastal water depends on many factors such as water flow and tidal differences. When the waters carrying sediment reach an area with lower tidal power and the sediment that has a more rapid falling speed tends to rest and deposit. Layers tend to differentiate according to sediment characteristics and components due to the condition that caused the deposit, such as size and density of the carried particles, and also tidal power [10-12].

Coastal area sediment deposits occur frequently in the form of mixed deposits and are referred to as graded sediments. This differs from the common conception that deposit is mostly found layered. This transformation of sediment form from layered to mixed can occur when the seabed is strongly pretreated. Mixing can also be a result of bioturbation: soil creatures living in coastal soil habitats as for example worms, dig the deposit layers and move them through ingestion and excretion from one level to the other, resulting in a mixed composition as a result of filter feeders [13-14]. This study proposes the analysis of sediments and rock specimens gathered from different areas of the Libyan eastern coast. The aims of the study can be briefly stated as follows: Utilizing Nanotechnology techniques (X–Ray Fluorescence) to evaluate Chemical Composition of Sediments and Rocks.

Methods

Sampling

Many sediment samples were collected from eight locations distributed along the far Eastern Libyan coast (near Tobruk region) from (Ras Al Tin village) to the east, to Ain El–Gazal area to the west (Table 1) during summer 2024. The collected samples were dried and kept until used.

Table 1. The sampled locations

Sample No	Sample Name
1	Ras Al –Tin
2	Enziwa
3	Al – Mongar
4	Al –Khabta
5	Al –Gifar
6	Al –Bomba
7	Al –Tamimy
8	Ain – AL gazala

Sample preparation

Collected samples were washed with distilled water, ground in a mortar, then garbled. According to several studies carried out on marine sediment samples [15-19].

Received*: 02-05-2025 -* ***Accepted****: 28-06-2025 -* ***Published****: 05-07-2025*

X-Ray Fluorescence scanning (XRF)

Specimen X-Ray and micro scanning were achieved by utilizing the X-Ray unit of Al-Fataieh Cement Factory, the central lab [20 -22].

Results

The studied samples composition of oxides are shown in Tables (2&3), the results showed that the present oxides were: (NaO, MgO, Al_2O_3, SiO_2, SO_3, CaO, & $CaCO_3$, Fe_2O_3, CuO, and ZnO) in the following percentages: (0.2, 0.7, 1.6, 0.2, 0.6, 11.1, 83.0, 0.1, 0.3 and 0.2 %), respectively, sediment sample details shown in Table (2). Conversely, the same elements oxides recorded for the Rock sample at the same location recorded concentrations as follows (0.1, 2.3, 1.2, 2.7, 0.6, 10.5, 79.7, 0.5, 0.6, and 0.4%). As an outcome of testing, it was found that the concentration of metal oxides in sediment samples and rocks sample at location (1) contain elevated levels of calcium carbonate ($CaCO_3$). While the metal oxides in sediments and rocks of sample (1) recorded (NaO, MgO, Al_2O_3, SiO_2, SO_3, CaO, CuO, ZnO, and Fe_2O_3) are extant a rate lower at location (4) in comparison with the carbonate in both sediment and rock samples. The trend distribution of oxides at this location is shown in order below:

$CaCO_3 >> CaO >> MgO > Al_2O_3 > SO_3$ *other oxides*

The results recorded by X–Ray analysis are in agreement with the chemical analysis outcomes, as both recorded high levels of calcium and carbonate in Sediments and Rocks samples.

Table 2. The concentrations of the studied metal oxides for samples (1 – 4).

Metal Oxide	1	2	3	4
SiO_2	1.46	0.50	1.30	0.87
Al_2O_3	0.43	0.22	0.44	0.30
Fe_2O_3	0.21	0.10	0.26	0.176
MgO	2.32	2.90	2.60	2.70
Na_2O	0.35	0.30	0.34	0.26
K2O	-	-	-	-
Cl	0.65	0.56	0.90	0.47
SO_3	1	1.14	1.17	0.97
TiO_2	-	-	-	-
MnO	0.01	0.01	0.01	0.01
P_2O_5	0.22	-	0.21	0.21
$CaCO_3$/CaO	89.23	90.1	87.60	90.76
$MgCO_3$	4.84	6.05	5.41	5.66

Table 3. The concentrations of the studied metal oxides in the samples (5 – 8).

Metal Oxide	5	6	7	8
SiO_2	1.20	0.40	0.33	1.11
Al_2O_3	0.35	0.22	0.30	0.53
Fe_2O_3	0.19	0.25	0.09	0.23
MgO	2.64	1.80	3.20	1.63
Na_2O	0.31	0.37	0.33	0.37
K2O	-	-	-	-
Cl	0.56	0.50	0.52	0.50
SO_3	1.04	0.90	1.14	0.92
TiO_2	-	-	-	-
MnO	0.01	0.02	0.011	0.02
P_2O_5	0.21	0.22	0.20	0.23
$CaCO_3$/CaO	90.38	92.30	89.41	91.30
$MgCO_3$	5.52	3.80	6.70	3.42

Discussion

The contents of metal oxides were illustrated in Tables 2 and 3, revealing some interesting patterns. At location 1, both sediments and rocks have relatively high levels of SiO_2, ranging from 1.7% to 2.7%. In contrast, the lowest concentration of SiO2 was found at location 4 in rocks and location 5 in sediments, with values of 0.1% and 0.3%, respectively. Interestingly, no significant presence of SiO_2 was detected at location 3 in rock samples. When it comes to Al_2O_3, high levels were obtained at location 3 in marine sediments and rocks, with concentrations of 1.9% and 3.1%, respectively. On the flip side, the lowest

Received: 02-05-2025 - **Accepted**: 28-06-2025 - **Published**: 05-07-2025

concentration of Al2O3 was observed at location 4 in rocks and location 3 in sediments, with values of 0.3% and 0.6%, respectively. Again, no significant presence of Al_2O_3 was detected at location 4 in sediment samples. For MgO, relatively high levels were obtained at station 1 in marine sediments and rocks, with concentrations of 2.9% and 2.3%, respectively. The lowest concentration of MgO was observed at location 2 in rocks and location 2 in sediments, with values of 0.3% and 0.6%, respectively.
High levels of SO_3 were recorded at location 3 in rocks and location 5 in sediments, with concentrations of 0.9% and 0.7 %, respectively. The lower contents of SO_3 were given was observed at location 5 in rocks and location 2 in sediments, with values of 0.4% and 0.4%, respectively.
When it comes to CaO, high levels were observed at location 3 in rocks and location 5 in marine sediments, with concentrations ranging from 11.8% to 11.1%, respectively. Conversely, low levels of CaO were observed at location 1 in rocks and the same was observed for sediments in location 2, with values of 10.5% and 10.3%, correspondingly. ($CaCO_3$) concentrations however were highest of all oxides, observed practically in location 4 in both sediment and rock, ranging from 84% to 783.8%, respectively. The lowest concentration of $CaCO_3$ was observed at location 3 in rocks and location 2 in sediment.
Lastly, the concentration of ZnO was generally lower than that of CuO at all locations, with high levels of ZnO observed at location 3 in rocks samples, and location 1in marine sediments are ranges (1.1 and 0.9 %) respectively, absence at location 4. By comparison the contents ZnO with that recorded for CuO at location 3 in rocks and location 1 in sediments were fluctuated in the ranges (2.2 – 1.0%) respectively. while was lower concentration observed at location 5 for CuO (0.5 and 0.1 %) in sediments and rocks.
Quartz is the main cause of AI and Si, however the presence of AI in the Xray examination is more likely due to the finding of clay minerals such as potash feldspar (KAlSiO8) and/or Kaolininte (Al2Si2O5),[23]. Dolomite on the other hand despite its occurrence in small amounts, could be the cause of Mg slight concentrations. The slight difference in concentrations of metal between locations is mainly because of the presence of the above minerals in variable concentrations, however X ray data was the identifier of oxides in the order displayed below

$CaCO_3 > CaO > SiO_2 > Al_2O_3 >>$ *other oxides*

As highlighted earlier, fine sediments, particularly clay particles, possess a remarkable ability to bind with various substances present in water. This binding process, referred to as sorption, involves two mechanisms: adsorption, where substances adhere to the surface of particles, and absorption, where substances are taken into the particles themselves. Such properties enable fine sediments to act as natural filters, removing dissolved contaminants from water. While this process reduces pollution levels in water, it simultaneously increases the contamination levels within the sediment bed. The partitioning of pollutants between their dissolved state in water and their binding to sediments is quantified by a parameter known as Kd. The value of this parameter is influenced by both the nature of the pollutant and the characteristics of the sediment. Laboratory studies have demonstrated that for inorganic contaminants, such as heavy metals like lead (Pb^{2+}), cadmium (Cd^{2+}), and copper (Cu^{2+}), the extent of partitioning is directly related to the sediment grain size. Finer grains, with their larger surface-area-to-volume ratio, exhibit stronger sorption capabilities, resulting in higher Kd values. Interestingly, this behavior is independent of the actual concentration of the dissolved heavy metals. However, a different dynamic applies to organic contaminants, Moreover, contaminants bound to sediments are considerably less toxic to marine organisms compared to their dissolved counterparts. Over time, as sediment beds age, the bioavailability of these pollutants diminishes further. Nonetheless, activities such as dredging or bioturbation can disrupt the sediment layers, introducing contaminants from deeper, oxygen-deficient layers into oxygen-rich environments, where desorption releases the attached metals back into the water. Conversely, burying sediments deeper into the soil can further reduce their bioavailability. Techniques such as the addition of activated carbon to soils have been found effective in minimizing the bioavailability of contaminants in sediment beds, thereby mitigating their ecological impact and is termed fluorescent radiation. Because the energy of the emitted photon is characteristic of the transition between specific electron orbitals in a particular element, the resulting fluorescent X-ray can be used to detect the abundances of elements that are present in the sample [23-24].
In most cases, for rocks, ores, sediments, and minerals, the sample is ground to a fine powder. At this point it may be analyzed directly in the case of trace element analyses. However, the very wide range of abundances of different elements, especially iron [25]. The studies of heavy metal contents in different marine samples as water, algae, sediments, or their treatments in different samples showed increasing of their contents due to human activities; therefore, the controlling the concentrations of heavy metals is very important [26-46].

Conclusion

This is which conducted on the sediment samples collected from the area extending from Ras Al–Tin village to Ain Gazalla region showed high values of Calcite (Calcium Carbonate mixed with small amounts of Dolomite ($CaMg(CO_3)_2$). The study also recorded small amounts of some metal oxides as NaO, MgO, Al_2O_3, SiO_2, Fe_2O_3, CuO, and MnO. Most of the detected oxides mainly coming from the geological sources of the studied area.

***Received**: 02-05-2025 - **Accepted**: 28-06-2025 - **Published**: 05-07-2025*

Conflict of interest. Nil

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***Received**: 02-05-2025 - **Accepted**: 28-06-2025 - **Published**: 05-07-2025*

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***Received**: 02-05-2025 - **Accepted**: 28-06-2025 - **Published**: 05-07-2025*