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Discovery of heavy mineral sand in Djiboutian coastline (Obock area, SE Afar rift)

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| <i>Keywords:</i> Djibouti Afar rift Heavy mineral sand Titanium dioxide | Background: In this paper, we present the first study of black sand deposits collected along the coastline of the Obock area, which is located in the North East part of Djibouti (SE Afar Rif). This area consists mainly of uplifted madrepores, sand dunes and alluvial deposits. Materials and Methods: Geochemical studies were carried out using techniques such as X-ray fluorescence (XRF) to quantify the heavy mineral sands. Electron microprobe and X-ray diffraction (XRD) techniques were used to identify minerals. Results: Mineral sands have been identified in coastal backshore deposits, containing light minerals (calcite, aragonite, quartz, anorthite, augite, microcline, albite, enstatite, nontronite and diopside) and heavy minerals (hematite, ilmenite, chromite and magnetite). In general, titanium dioxide (TiO₂) and iron oxide (Fe₂O₃) are found in the backshore, with averages of TiO₂ (23.9 %) and Fe₂O₃ (53.9 %) in the total of heavy mineral fraction. Conclusions: Although, titanomagnetite is not widely exploited in the world due of the treatment to enrich ores with Fe and Ti, the presence of ilmenite and vanadium as trace elements in the subsurface and in titanomagnetite | | | | | | | |
| | respectively, even at very low grades may potentially justify further of the lateral extent and depth geochemical and drilling investigations in order to upgrade the concentration of titanium oxide in the Obock coastal sands. | | | | | | | |

1. Introduction

The Republic of Djibouti is located at the intersection of two oceanic structures, the Red Sea and the Gulf of Aden, and the continental East African Rift System (EARS). It benefits from a unique geodynamic position (Fig. 1a). This small country has been extensively studied from a petrological, geophysical and structural point of view [1–4]. Hydrothermal ore deposits have been explored and epithermal gold deposits have been described in several areas, mostly associated with acidic intrusions [5–7]. These occurrences are mainly represented by base and precious metal bearing quartz/carbonate adularia veins.

Heavy mineral sand (HMS) deposits play an important role in coastal environments and are the world's economic source of titanium minerals [9–22]. These types of deposits, mainly hosted in the sand fraction, can also be occur in the silt and clay fractions with economic concentrations of heavy minerals [21]. Coastal deposits with high concentrations of heavy minerals can be mined, recovered, and used in a wide range of

industrial mineral applications. Titanium is a transition metal that is used in industries such as aerospace and bio-medical for its high resistance to corrosion, high refractive index and ability to absorb UV light. It is also commonly used in the pigment industry. Titanium occurs in the Earth's crust in two main minerals: ilmenite (FeTiO₃) and rutile (TiO₂). The alteration of igneous and metamorphic rocks is largely responsible for the concentration of HMS in the coastal environments. Many authors [19,21,22] have explained that their deposition is mainly due to natural processes such as weathering, followed by erosion, transport, and sedimentation. The main agents of transport are water, wind and ice, and gravity, or simply the difference in specific gravity of the minerals applied to the particles being transported [21]. These particles are transported by rivers and then accumulate in various coastal environments such as deltas, coastlines, beaches, tidal lagoons and floodplains. The sediments are reworked by wind, waves, and tides, which separate them by density and size. These processes allow the formation of laminated deposits with alternating black and white lines or dispersing them

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Fig. 1. a) Map of Africa. b) Geological map of the Republic of Djibouti showing and the location of the sampled areas (c) (purple squares) [8].

in the sand. HMS have been reported worldwide from the Cretaceous to the Holocene [21].

In this article, we report the first geochemical and mineralogical study of the black sand from the Obock region (northeast of the Republic of Djibouti) to determine the presence of titanium oxide, which is considered as valuable heavy mineral (VHM) in the world. In this study, geochemical and mineralogical investigations were carried out using X-ray diffraction, electron microprobe and X-ray fluorescence techniques to identify the composition of rocks and associated minerals. The discovery of HMS is very important for country's economy by generating foreign investment and creating related industries, which must also be well managed to prevent environmental and social impacts.

2. Climatology setting

The Republic of Djibouti is a small country $(23,200 \text{ km}^2)$ situated in the Horn of Africa that experiences extreme heat conditions and low rainfall, with annual cumulative rainfall ranging from 60 to 300 mm,

with an average of around 150 mm [23]. The northeastern coastal plains are the least watered area of Djibouti, with annual rainfall ranging from 50 to 100 mm [24]. The rains are often unpredictable and sparse, leading to frequent droughts. Djibouti's climate is classified as arid according to the Köppen-Geiger climate classification (climate type code: BWh; [25]). Djibouti has two seasons: a cool season from October to April and a hot season from May to September. Northeasterly trade winds from Saudi Arabia and the Gulf of Aden dominate the winter climate, with average temperatures of 30 °C. Equatorial westerly winds dominate the summer, with average temperatures between 30 °C and 45 °C. Djibouti has a high evapotranspiration with an annual rate of 2000 mm (BGR, 1982).

Due to the country's arid climate, most watercourses in the Republic of Djibouti are only operational for a few hours after a rainfall event (wadis) [26,27]. A hot and dry westerly wind regime, that reduces visibility and contributes to dust storms, known as khamsin, is caused by the warming and drying of the East African monsoon, also known as the foehn effect, occurs as the wind blows across the Ethiopian and Somali mountain ranges. This exacerbates the aridity of the climate. The local climate is influenced by the country's complex topographical features and its proximity to the Indian Ocean [24]. Variations in rainfall in Djibouti are a result of large-scale climate variability, specifically linked to the Indian Ocean Dipole and the El Niño–Southern Oscillation [23, 26].

3. Geological setting

The first evidence of synrift magmatism in Djibouti are the 28–20 Ma Ali Sabieh mafic series overlying Meso-Cenozoic sedimentary substratum terranes in the Ali Sabieh antiformal area [28] and the more extensive 19–11 Ma Mablas felsic lavas and finally the 8.6–3.8 Ma Dalha basalts. At 3 Ma, a major kinematic change occurred in the Afar rift system, as evidenced by: (i) the emplacement of the 3–1 Ma stratoid trap-like basalts that floored the Afar Triangle, and (ii) the westward propagation of the Gulf of Aden accretionary axis through the Afar rift crust [4,29] via the Tadjoura rift [30] and its onshore extension to the NW along the Asal axis (Fig. 1b). The Asal Rift axis is considered as a nascent oceanic ridge [2,31,32].

The area studied is located along the 235 km northeast region of the Republic of Djibouti (Fig. 1c), on the western coast of the Bab-el-Mandeb Strait, Obock is a region well known for its exceptional landscapes composed of mangroves, marine remains and volcanic islands (Sawabi chains). This area contains both marine and continental remains, consisting of (i) fluvial alluvium near rivers and coastal deposits of Holocene age; (ii) marine deposits of Upper Pleistocene age [33]. Volcanic formations composed of the Mablas, Dahla and the Quaternary Sawabi chains are encountered in this area. The Mablas rhyolitic series correspond to a vast series of dome and lava flows dated between 19–11 Ma, present in two sectors, on either side of the Gulf of Tadjoura. They unconformably overlies the Dahla basaltic series (8.6–3.8 m.y.), which form the outer margin of the Ali Sabieh antiform and the western flank of the Danakil depression [31].

The geology of Obock is dominated by sedimentary formations, including the first generation of reef limestones, dated to 300–250 Ka [33,34], which form a vast plateau. The second generation of reef outcrops at the base of the first formation $(130 \pm 11 \text{ Ka}; [33,34])$. The Mabla rhyolitic massif (Miocene) dominates the relief, which is cut by deep and narrow wadis that follow the major faults. This region allows the observation of the onshore extension of recent faults, that characterize the activity of the submarine segment of the ridge [3]. This extension also explains the significant uplift of the coral reefs [33,35]. The area also has hot springs along the coast and its potential as a geothermal resource has been discussed in [36] and [37].

Geomorphologically, the entire coastline of the Obock area is not homogeneous. Most of the wadis (Inda-Tagarre, Sadai, Debergadé) and their tributaries originate at the foot of the old Mablas rhyolites and

Dahla basalts, flowing into the sea and helping to bring placer minerals. The coastline is dominated by sedimentary formations, including the first generation of reef limestones, which form a vast plateau with 50 m high plateaus (Baradle, Gahro). The second generation of reef outcrops at the foot of the first formation (m2; 130 ± 11 Ka; [33,34]). They form the Bagainda and Moudaoudli plateaus around 25 m above sea level while conglomeratic fluvial deposits (100 to 10 Ka) with 10 m thick cover the highest reef plateaus [3]. In the southwest of Obock, the rhyolitic Mabla montains are separated from the sea by a 3 km-wide, coastal strip and the reefs cap the Gulf basalts (1.5 Myr old). In the north, thick, coarse conglomeratic deposits (QA) are interbedded between the basalts and the reefs. These fluvial alluvial deposits crossed by the Debergadé paleo-valley consist of large-block conglomerates, up to several tens of meters thick, derived from the intensely altered reliefs of Mablas rhyolites and Dahla basalts [3]. The rhyolitic gravels of the Mablas series, have been uplifted in response to Quaternary climate change, form a reg on the surface of the coral plateaus.

4. Materials and methods

4.1. Sampling

In this study, we sampled at the backshore area because that appeared visually darker and more concentrated in heavy minerals compared to other areas, including the frontshore. Fifteen representative samples of black sand were collected manually along the backshore of the South and North of the Obock region (Fig. 1c). Sampling was carried out in three different areas. The first area is located in the South of Obock, between Tadjoura and Obock, where nine samples were collected between N11°.57.450'; E 43°.12.681' for DJ-OB-52 and N11°.51.409'; E 43°05.774' for DJ-OB-65; in an area of 12 km length and approximately 50 m wide on the backshore. The area is characterized by different deposits such calcareous reef, sand dune and sand. This location is shown in Fig. 2a. The second area is located between Obock and Godorya beach, corresponding to the location of N12°09.201' and E 43°24.836' for the sample DJ-GD-02. The Godorya site is separated of Obock beach by a large steep cliff where sampling was not possible. This area is about 40 km from the town of Obock. Godorya is well known for its dense mangrove. The sample was taken from the backshore of the beach (Fig. 2e). The third area which includes essential facies of sand dune, marine and alluvial deposits was sampled between Godorya and Khor Angar which correspond to between N12°16.947' and E 43°.22.317'; N12°16.839' and E 43°22.693' for the six samples between DJ-K-01 and DJ-K-33. All three areas were sampled from the subsurface at depths between 5-20 cm using hand tools like shovels. The samples showed alternate dark black and brownish white bands (Fig. 2c e) with thickness varies from 1 to 3 cm. Each sample was packed in a bag and labelled accordingly for laboratory analyses.

4.2. Analytical methods

Samples from the Obock area were analyzed at the ALS Metallurgy



Fig. 2. a, b) examples of field photos showing a black sand in a beach or dune (respectively) south of Obock; c) examples of field site showing 40 cm deep layers of black sand (south of Obock); d) examples of black sand deposits associated with paleo-beach; e, f) example of field photos of black sand deposits.

Laboratory in Australia using (i) heavy liquid (tetrabromoethane) with a density of 2.96 g/cm³ for magnetic separation and (ii) XRF elemental analysis for major and trace element composition using the XRF MS method; routine major and some trace elements were measured on samples prepared as glass discs (SiO₂, Al₂O₃, Fe₂O₃, MgO, CaO, Na₂O, K2O, TiO2, P2O5, Mn3O4, Cr2O3, Hf, CeO2, La2O3, Th, SnO2 SO3, U and ZrO₂) before and for some elements after magnetic separation (Table 1). A total of eight geological certified reference materials (CRMs) were analyzed, including iron ore samples (GIOP 17, GIOP-115, GIOP-55, GIOP-15, GIOP-33), phosphate rock, and titanoferous-vanado-magnetite (NCS DC 79,002, CSIRO 243, AMIS 0347), as well as five in-house reference materials (RMs) containing zircon and ilmenite sand (AA-ILM 001, AA-ZIR 003, AA-TMS17-MSHigh, AA-ILM 002, AA-ZIR 005) during the analytical session. The analytical precision was <10 % relative standard deviation (RSD) for major element concentrations greater than 0.01 % and trace element abundances greater than 0.0004 %. The accuracy was within 10 % for major element concentrations above 0.1 wt % and for trace element concentrations greater than 0.004 %. The flow chart is illustrated in Fig. 3.

Samples of the heavy mineral concentrate were subjected to magnetic separation at 6000 Gauss using a metal disc magnet. The sample was fed from a vibrating feeder onto a conveyor belt, which passed under a rotating disc with an induced magnetic field. Magnetic material was lifted onto the disc and then discharged into a collection chute as the disc rotated out of the magnetic field. Non-magnetic material is collected in a hopper at the end of the conveyor. The magnetic and non-magnetic fractions are then collected and weighed. The test work is carried out as follows: a 100-200 g sample is split using a riffle splitter and the appropriate density heavy liquid is prepared by either evaporation or dilution of the concentrate. The density is checked with a hydrometer. Then, the heavy liquid is added to a separating funnel. The sample is added to the funnel and stirred to ensure wetting of the surfaces (air bubbles will result in dense particles floating). Samples are allowed to separate for several minutes. The layer that floats is stirred once more to release any that may be trapped and the particle that sinkare drained through the valve at the bottom of the separating funnel and washed with an appropriate solvent. The floats are recovered, filtered, washed with an appropriate solvent, and discarded. The sinks are dried at 105 °C and weighed. The drying process removes any residual organic heavy liquids.

The magnetic and non-magnetic samples were analyzed by X-ray diffraction (XRD) at CERD's metallogeny laboratory to determine phase minerals. XRD analyses were performed using CuK α radiation on a Bruker D8 instrument. Powder samples were step-scanned from 10 to 70° at 0.02°, 20 intervals for a time period of 0.5 s. In addition, X-ray elemental mapping was performed using a CAMECA SX100 electron microprobe at "Microsonde Ouest" (Ifremer, France) with the PAP correction program (Pouchou and Pichoir, 1984), under operating conditions of 25 kV acceleration voltage, 20 nA beam current and a 1 μ m² beam size.

5. Results

5.1. Mineralogical analysis

The main minerals identified in the initial bulk samples by XRD at Obock, Khor Angar and Godorya are calcite, aragonite, quartz, anorthite, augite, microcline, grossular, diopside, pigeonite, albite, enstatite, nontronite, glauconite, hematite, chromite, ilmenite, magnetite and traces of cobalt associated with iron.

Magnetite, ilmenite, hematite and chromite were also identified at the three sites. Additionally, mapping by electron microprobe was carried out to determine the Ti and Fe at the grain scale (10 wt % for the d-e and around 60 wt % for j - k images) (Fig. 4). The X-ray maps and the black-scattered images show that high silicate and carbonate grains (calcite) are also present (Fig. 4). Microprobe analysis revealed the

| Sample ID | | | | | | | | OD CL | 20.00 | 17 0.1 | | V 16 /1 | V 0 V | 1 00 1 | |
|-------------------------------------|---------|---------|--------|---------|--------|---------|--------|--------|---------|---------|---------|---------|--------|--------|---------|
| an and much | OB-52 | OB-56 | OB-58 | OB-59 | OB-62 | OB-62-A | OB-64 | C0-90 | OB-66 | K-01 | K-04 | V-17-V | N-24 | K-33-A | GD-02 |
| Mass to HLS (g) | 199.17 | 199.78 | 197.87 | 198.66 | 198.49 | 197.56 | 198.39 | 196.97 | 198.44 | 198.81 | 198.68 | 198.94 | 195.79 | 169.15 | 198.57 |
| Mass Float | 28.28 | 19.82 | 106.61 | 14.57 | 38.5 | 12.45 | 105.39 | 145.36 | 72.92 | 43.01 | 27.88 | 34.73 | 70.81 | 9 | 159.74 |
| % Float | 14.20 | 9.92 | 53.88 | 7.33 | 19.40 | 6.30 | 53.12 | 73.80 | 36.75 | 21.63 | 14.03 | 17.46 | 36.17 | 3.55 | 80.45 |
| Mass HMC | 170.89 | 179.96 | 91.26 | 184.09 | 159.99 | 185.11 | 93.00 | 51.61 | 125.52 | 155.80 | 170.80 | 164.21 | 124.98 | 163.15 | 38.83 |
| % HMC | 85.80 | 90.08 | 46.12 | 92.67 | 80.60 | 93.70 | 46.88 | 26.20 | 63.25 | 78.37 | 85.97 | 82.54 | 63.83 | 96.45 | 19.55 |
| Al ₂ O ₃ (%) | 3.14 | 3.4 | 4.81 | 3.92 | 3.61 | 2.4 | 4.12 | 4.32 | 3.41 | 2.78 | 3.03 | 2.82 | 3.05 | 2.20 | 3.55 |
| CaO(%) | 5.18 | 6.11 | 9.25 | 7.24 | 5.68 | 2.90 | 7.85 | 8.53 | 7.27 | 9.59 | 13.1 | 9.73 | 11.0 | 6.77 | 15.7 |
| CeO ₂ (%) | 0.01 | < 0.01 | < 0.01 | <0.01 | <0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | <0.01 | < 0.01 | < 0.01 | < 0.01 |
| Cr ₂ O ₃ (%) | 0.12 | 0.12 | 0.12 | 0.13 | 0.12 | 0.14 | 0.12 | 0.11 | 0.12 | 0.14 | 0.12 | 0.14 | 0.13 | 0.16 | 0.13 |
| Fe ₂ O ₃ (%) | 48.6 | 46.1 | 34.1 | 40.5 | 43.9 | 53.9 | 39.7 | 36.4 | 42.9 | 38.9 | 32.0 | 36.5 | 29.4 | 45.9 | 21.0 |
| SiO2(%) | 18.1 | 20.8 | 31.3 | 23.8 | 20.1 | 11.0 | 26.7 | 28.7 | 23.5 | 22.0 | 24.0 | 23.3 | 28.8 | 14.5 | 37.2 |
| TiO ₂ (%) | 17.2 | 15.4 | 10.3 | 15.5 | 18.2 | 23.9 | 12.1 | 11.6 | 14.1 | 17.6 | 14.0 | 16.5 | 12.3 | 22.1 | 8.63 |
| MgO(%) | 4.74 | 5.43 | 7.50 | 5.94 | 4.75 | 3.54 | 6.62 | 6.98 | 6.35 | 6.59 | 6.93 | 7.06 | 8.51 | 4.82 | 10.3 |
| Mn ₃ O ₄ (%) | 0.70 | 0.65 | 0.51 | 0.64 | 0.70 | 0.85 | 0.54 | 0.51 | 0.58 | 0.61 | 0.51 | 0.58 | 0.47 | 0.68 | 0.36 |
| P ₂ O ₅ (%) | 0.13 | 0.14 | 0.19 | 0.15 | 0.14 | 0.11 | 0.16 | 0.17 | 0.14 | 0.10 | 0.11 | 0.10 | 0.10 | 0.087 | 0.12 |
| $La_2O_3(\%)$ | 0.01 | < 0.01 | 0.01 | < 0.01 | <0.01 | <0.01 | 0.01 | <0.01 | <0.01 | 0.01 | < 0.01 | 0.01 | <0.01 | 0.01 | < 0.01 |
| Hf(%) | 0.002 | 0.001 | 0.001 | 0.003 | 0.003 | 0.005 | 0.003 | 0.002 | <0.001 | 0.002 | 0.002 | 0.004 | 0.003 | 0.002 | < 0.001 |
| SnO ₂ (%) | < 0.004 | < 0.004 | <0.004 | <0.004 | <0.004 | <0.004 | <0.004 | <0.004 | <0.004 | < 0.004 | < 0.004 | <0.004 | <0.004 | <0.004 | 0.005 |
| SO ₃ (%) | 0.059 | 0.023 | 0.048 | 0.058 | 0.057 | 0.064 | 0.038 | 0.057 | 0.028 | 0.085 | 0.14 | 0.098 | 0.25 | 0.058 | 0.088 |
| Th(%) | 0.002 | < 0.001 | <0.001 | < 0.001 | <0.001 | < 0.001 | 0.002 | <0.001 | < 0.001 | < 0.001 | < 0.001 | < 0.001 | 0.002 | <0.001 | 0.002 |
| N(%) | < 0.001 | < 0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | 0.001 | 0.002 | < 0.001 | < 0.001 | <0.001 | <0.001 | <0.001 | < 0.001 |
| ZrO ₂ (%) | 0.11 | 0.10 | 0.07 | 0.15 | 0.15 | 0.20 | 0.08 | 0.08 | 0.09 | 0.09 | 0.07 | 0.08 | 0.07 | 0.13 | 0.06 |
| TOI (%) | 0.33 | 0.37 | 0.87 | 0.89 | 0.96 | -0.21 | 0.86 | 1.23 | 0.64 | 1.72 | 5.17 | 2.09 | 4.30 | 1.17 | 2.13 |
| Total (%) | 99.77 | 99.73 | 100.50 | 100.37 | 99.98 | 100.21 | 100.16 | 100.03 | 100.24 | 101.44 | 100.76 | 100.35 | 101.74 | 99.60 | 100.12 |



Fig. 3. Flow chart diagram of the black sands.

presence of iron oxide, diopside, and ilmenite with 53 % and 44.6 % for TiO_2 and FeO, respectively, with traces of magnesium, aluminum, manganese and vanadium (~1 wt %). Titanomagnetite was also detected with FeO at 64.6 wt % and TiO_2 at 22 wt %. Mapping of the two samples shows that iron and titanium are about 52 wt % and 27 wt % respectively.

5.2. Major element concentrations

The major element concentrations of the samples analyzed by XRF are listed in Table 1. At the Godorya site, the concentrations of TiO_2 and Fe_2O_3 are 8.6 % and 21 % respectively in the samples while SiO_2 concentration is 37.6 % and CaO range from 15.7 %. The Al_2O_3 shows a value of 3.5 %.

The Khor Angar area is characterized by TiO₂ with values between 12.3 % and 22.1 % and Fe₂O₃ concentration ranging from 29.4 % and 45.9 % in the different samples. The SiO₂ concentration falls between 14.5 % to 28.8 % and CaO ranges from 6.7 to 13.1 %. The Al₂O₃ concentration yield between 2.2 and ~3 %.

The Obock site exhibit the highest TiO₂ and Fe₂O₃ values, which correspond to 10.3 % to 24 % and 34.1 % to 53.9 % respectively (Table 1). The SiO₂ concentration is between 11 % and 28.7 % while CaO range from 2.9 % to 9.2 %. The Al₂O₃ concentration is between 2.4 % to 4.8 %. In Additionally, some minor elements have been detected as ZrO_2 (with a maximum value of 0.20 %).

5.3. Magnetic and non-magnetic element concentrations

The major element concentrations of the magnetic and non-magnetic samples are listed in Table 2. Thirteen out of the 15 samples had mass pulls of 62 %–87 % to the magnetic fraction. Consequently, there was little improvement in the concentration of valuable mineral with the highest TiO₂ grades reaching only ~26.5 % (Table 2). The grades of valuable heavy mineral (VHM) were relatively low with TiO₂ concentration of the HMC (heavy mineral composite) ranging from 8.6–24 %. The zircon grades show a value of 0.20 %. The concentration of Th, U, La, and Ce were all essentially at or below their respective detection limits. The concentrates also contained relatively high concentrations of silicate with combined Al₂O₃, CaO, MgO, and SiO₂ typically in the 30 %–40 % range.

At the Godorya site, the TiO_2 content ranges from 3.5 % and 4.2 % while Fe_2O_3 are between 13.3 % and 16.3 % in the samples. The SiO_2 concentration is 22 % and CaO has a value of 7.05 % and Al_2O_3 shows values of 3.6 %.

The Khor Angar area is characterized by TiO₂ values ranging from 16.3 % to 26.4 %, and Fe₂O₃ concentrations ranging from 16.3 % to 26.4 % in the different samples. The SiO₂ concentration is between 9.7 % and 16.7 % and CaO range from 4.4 % to 3.1 %. The Al₂O₃ concentration yield between 2.5 % and 2.9 %.

The Obock area exhibits the highest TiO_2 and Fe_2O_3 values, corresponding to 14.5 % to 26.3 % and 47.30 % to 56.1 % respectively (Table 2). The SiO₂ concentration is between 12.9 % and 25.7 %, while CaO ranges from 1.9 % to 6.6 %. The Al_2O_3 concentration is between 3.2 % to 5.29 %.



Fig. 4. Electron Microprobe photos of samples from Obock area with X-ray maps of Si-Ca- Al Kα lines showing the presence of silicate and carbonate minerals for a, b, c, g, h, j images; and for d, e, j and k images X-ray maps Kα lines; f and l are the black scattered electron images showing the presence of titanium and iron grains in the samples.

6. Discussions

6.1. Origin of the black sands

The concentration of titanium dioxide in the basalt (Dahla) and rhyolite (Mablas) of the Obock area ranges from 1.50 % to 3.54 % representing the typical concentration found in this type of rock in the worldwide [35]. The main minerals present in the samples are

associated with volcanic rocks. Calcite and aragonite are carbonate minerals and likely associated with the madrepore deposits. Other minerals found in the area include quartz, anorthite, augite, microcline, grossular, diopside, pigeonite, albite, enstatite, nontronite, glauconite, hematite, chromite, ilmenite and magnetite, all resulting from the alteration of basalts. The black sand in the backshore environment extend for about 12 km in the southern region of Obock. The coastline of the Obock area is characterized by several wadis (Inda-Tagarre, Sadai,

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Table 2 Results of the analyzed magnetic and non-magnetic samples by XRF.

| Sample ID | | Mass (g) | % | Al_2O_3 | CaO | CeO ₂ | Cr_2O_3 | Fe ₂ O ₃ | Fe ₂ O ₃ Dist % | Hf | La_2O_3 | MgO | Mn ₃ O ₄ | P_2O_5 | SiO_2 | $\rm SiO_2$ Dist % | SnO_2 | SO_3 | Th | TiO ₂ | TiO ₂ Dist % | U | ZrO_2 |
|-----------------|---------------------------|-------------|--------------|------------|--------------|------------------|-----------|--------------------------------|---------------------------------------|---------|-----------|--------------|--------------------------------|----------|--------------|-------------------------|------------------|--------|---------|------------------|-------------------------|---------|---------|
| DJ_OB 52 2020 | MAG | 79.01 | 76.16 | 3.3 | 3.28 | < 0.01 | 0.11 | 52.5 | 85.13 | 0.006 | < 0.01 | 3.55 | 0.8 | 0.15 | 14.2 | 55.69 | < 0.004 | 0.062 | 0.001 | 20.3 | 94.76 | < 0.001 | 0.09 |
| | NONMAG | 24.73 | 23.84 | 3.49 | 12.8 | < 0.01 | 0.16 | 29.3 | 14.9 | 0.004 | < 0.01 | 9.39 | 0.33 | 0.07 | 36.1 | 44.3 | < 0.004 | 0.049 | 0.002 | 3.59 | 5.24 | < 0.001 | 0.19 |
| | Calculated Head | - | - | 3.35 | 5.55 | < 0.01 | 0.12 | 47.0 | - | 0.01 | < 0.01 | 4.94 | 0.69 | 0.13 | 19.4 | - | < 0.004 | 0.059 | 0.001 | 16.2 | - | < 0.001 | 0.11 |
| DJ_OB 56 2020 | MAG | 70.94 | 74.56 | 3.26 | 3.48 | < 0.01 | 0.11 | 51.7 | 84.2 | 0.004 | < 0.01 | 3.76 | 0.78 | 0.15 | 14.7 | 53.7 | < 0.004 | 0.018 | < 0.001 | 20.1 | 94.74 | < 0.001 | 0.09 |
| | NONMAG | 24.21 | 25.44 | 3.5 | 13.3 | 0.01 | 0.18 | 28.4 | 15.8 | 0.003 | 0.01 | 10.3 | 0.31 | 0.071 | 37.2 | 46.3 | < 0.004 | 0.022 | 0.001 | 3.3 | 5.26 | < 0.001 | 0.15 |
| | Calculated Head | - | - | 3.32 | 5.98 | < 0.01 | 0.13 | 45.8 | _ | 0.00 | < 0.01 | 5.42 | 0.66 | 0.13 | 20.4 | _ | < 0.004 | 0.019 | < 0.001 | 15.8 | - | < 0.001 | 0.11 |
| DJ_OB 58 2020 | MAG | 54.69 | 63.85 | 5.29 | 6.16 | < 0.01 | 0.088 | 38.9 | 76.9 | 0.003 | < 0.01 | 5.37 | 0.61 | 0.26 | 25.7 | 51.64 | < 0.004 | 0.054 | 0.002 | 14.5 | 91.66 | < 0.001 | 0.07 |
| | NONMAG | 30.97 | 36.15 | 4.02 | 15.2 | < 0.01 | 0.19 | 20.7 | 23.16 | 0.004 | < 0.01 | 11.7 | 0.29 | 0.079 | 42.5 | 48.36 | < 0.004 | 0.033 | 0.002 | 2.3 | 8.34 | < 0.001 | 0.07 |
| | Calculated Head | - | - | 4.83 | 9.43 | < 0.01 | 0.12 | 32.3 | - | 0.003 | < 0.01 | 7.66 | 0.49 | 0.19 | 31.8 | - | < 0.004 | 0.05 | 0.002 | 10.1 | - | < 0.001 | 0.07 |
| DJ_OB 59 2020 | MAG | 73.4 | 72.09 | 3.0 | 3.83 1E 4 | < 0.01 | 0.15 | 46.6 | 80.3 12.9 | 0.002 | < 0.01 | 4.05 | 0.79 | 0.18 | 10.2 | 50.57 40.42 | < 0.004 | 0.050 | < 0.001 | 20.5 | 93.99 | < 0.001 | 0.09 |
| | Colculated Head | 20.42 | 27.91 | 3.84 | 7.06 | < 0.01 | 0.13 | 20.1 | 13.8 | 0.003 | <0.01 | 5.82 | 0.5 | 0.000 | 40.9 | 49.43 | < 0.004 | 0.056 | < 0.001 | 3.4 15.6 | 0.01 | <0.001 | 0.27 |
| DI OB 62 2020 | MAG | 78.82 | 76.01 | 3.12 | 2.89 | < 0.01 | 0.14 | 51.8 | 88.1 | 0.004 | < 0.01 | 3.52 | 0.03 | 0.15 | 12.9 | 51 17 | < 0.004 | 0.030 | < 0.001 | 23.2 | 94 24 | < 0.001 | 0.14 |
| DU_0D 02 2020 | NONMAG | 24.88 | 23.99 | 4.73 | 13.5 | 0.02 | 0.13 | 22.1 | 11.9 | 0.007 | 0.01 | 8.54 | 0.3 | 0.089 | 39 | 48.83 | 0.005 | 0.088 | < 0.001 | 4.5 | 5.76 | < 0.001 | 0.33 |
| | Calculated Head | _ | _ | 3.51 | 5.44 | < 0.01 | 0.13 | 44.67 | _ | 0.005 | < 0.01 | 4.72 | 0.71 | 0.14 | 19.2 | _ | < 0.004 | 0.055 | < 0.001 | 18.7 | _ | < 0.001 | 0.16 |
| DJ OB 62/A 2020 | MAG | 90.58 | 87.45 | 2.27 | 1.87 | < 0.01 | 0.15 | 56.1 | 91.9 | < 0.001 | 0.01 | 2.95 | 0.93 | 0.12 | 8.4 | 67.41 | < 0.004 | 0.059 | < 0.001 | 26.3 | 95.50 | 0.001 | 0.11 |
| | NONMAG | 13 | 12.55 | 3.02 | 9.78 | 0.03 | 0.15 | 34.3 | 8.1 | 0.010 | 0.02 | 7.59 | 0.4 | 0.089 | 28.3 | 32.59 | 0.005 | 0.11 | 0.002 | 8.6 | 4.50 | 0.001 | 0.76 |
| | Calculated Head | _ | _ | 2.36 | 2.86 | < 0.01 | 0.15 | 53.4 | - | 0.001 | 0.01 | 3.53 | 0.86 | 0.12 | 10.9 | _ | < 0.004 | 0.065 | < 0.001 | 24.1 | - | 0.001 | 0.19 |
| DJ_OB 64 2020 | MAG | 63.5 | 72.51 | 4.14 | 4.66 | < 0.01 | 0.091 | 47.3 | 85.9 | 0.004 | < 0.01 | 4.5 | 0.67 | 0.2 | 19.5 | 54.82 | < 0.004 | 0.039 | 0.003 | 16.1 | 94.23 | 0.001 | 0.08 |
| | NONMAG | 24.07 | 27.49 | 3.85 | 15.5 | < 0.01 | 0.2 | 20.4 | 14.1 | 0.004 | < 0.01 | 11.7 | 0.24 | 0.074 | 42.4 | 45.18 | < 0.004 | 0.04 | < 0.001 | 2.6 | 5.77 | < 0.001 | 0.11 |
| | Calculated Head | - | - | 4.06 | 7.64 | < 0.01 | 0.12 | 39.9 | - | 0.004 | < 0.01 | 6.48 | 0.55 | 0.17 | 25.8 | - | < 0.004 | 0.039 | 0.002 | 12.4 | - | 0.001 | 0.09 |
| Sample ID | | Mass (g) | % | Al_2O_3 | CaO | CeO ₂ | Cr_2O_3 | Fe ₂ O ₃ | Fe ₂ O ₃ Dist % | Hf | La_2O_3 | MgO | Mn_3O_4 | P_2O_5 | SiO_2 | SiO ₂ Dist % | SnO_2 | SO_3 | Th | TiO ₂ | TiO ₂ Dist % | U | ZrO_2 |
| DJ_OB 65 2020 | MAG | 31.7 | 68.9 | 4.23 | 5.31 | < 0.01 | 0.089 | 44 | 84.2 | 0.002 | 0.01 | 5.14 | 0.63 | 0.21 | 21.9 | 52.78 | < 0.004 | 0.063 | 0.001 | 15.2 | 92.65 | < 0.001 | 0.07 |
| | NONMAG | 14.33 | 31.10 | 4.29 | 15.8 | < 0.01 | 0.17 | 18.3 | 15.8 | < 0.001 | < 0.01 | 11.4 | 0.24 | 0.087 | 43.4 | 47.2 | < 0.004 | 0.043 | < 0.001 | 2.67 | 7.35 | < 0.001 | 0.09 |
| DI OD (C DODO | Calculated Head | - | - | 4.25 | 8.57 | < 0.01 | 0.11 | 36.0 | - | 0.001 | 0.01 | 7.09 | 0.51 | 0.17 | 28.6 | - | < 0.004 | 0.06 | 0.001 | 11.3 | - | < 0.001 | 0.08 |
| DJ_OB 66 2020 | MAG | 71.4 | 72.5 | 3.3 | 3.97 | < 0.01 | 0.096 | 51.1 | 86.2 | 0.006 | < 0.01 | 4.31 | 0.71 | 0.16 | 16.4 | 50.91 | < 0.004 | 0.030 | < 0.001 | 18.1 | 94.1 | < 0.001 | 0.08 |
| | NONMAG Colgulated Hood | 27.1 | 27.5 | 3.05 | 15.5 | 0.02 | 0.19 | 21.0 | 13.84 | 0.006 | < 0.01 | 11./ 6.2E | 0.25 | 0.071 | 41.0 | 49.09 | < 0.004 | 0.025 | 0.002 | 3 | 5.93 | 0.001 | 0.12 |
| DIK 01 2020 | MAG | - | - 70.1 | 2.54 | 7.13 4.42 | < 0.01 | 0.12 | 48.5 | - 88.2 | 0.000 | <0.01 | 4 74 | 0.38 | 0.14 | 23.3 14.6 | - 46 5 | < 0.004 | 0.029 | 0.001 | 23.54 | - 03.7 | <0.001 | 0.09 |
| DJ_R 01 2020 | NONMAG | 29.5 | 29.9 | 3.34 | 21.1 | < 0.01 | 0.13 | 14.2 | 11.09 | 0.002 | 0.01 | 11.1 | 0.28 | 0.05 | 39.5 | 53.55 | < 0.004 | 0.11 | < 0.001 | 3.68 | 6.26 | < 0.001 | 0.05 |
| | Calculated Head | - | _ | 2.8 | 9.40 | 0.00 | 0.14 | 38.25 | _ | 0.002 | 0.00 | 6.64 | 0.61 | 0.10 | 22.0 | _ | < 0.004 | 0.08 | 0.001 | 17.58 | - | < 0.001 | 0.09 |
| DJ K 04 2020 | MAG | 61.2 | 62.5 | 2.8 | 5.24 | < 0.01 | 0.13 | 45.6 | 86.6 | 0.003 | < 0.01 | 5.17 | 0.71 | 0.13 | 16.7 | 43.76 | < 0.004 | 0.08 | < 0.001 | 21.5 | 92.8 | < 0.001 | 0.09 |
| | NONMAG | 36.7 | 37.5 | 3.3 | 24.2 | < 0.01 | 0.12 | 11.8 | 13.43 | < 0.001 | < 0.01 | 10 | 0.23 | 0.051 | 35.8 | 56.24 | < 0.004 | 0.17 | < 0.001 | 2.8 | 7.24 | 0.001 | 0.06 |
| | Calculated Head | - | _ | 2.95 | 12.35 | < 0.01 | 0.13 | 32.9 | _ | 0.002 | < 0.01 | 6.98 | 0.53 | 0.10 | 23.9 | _ | < 0.004 | 0.11 | < 0.001 | 14.49 | _ | < 0.001 | 0.08 |
| DJ_K 21/A 2020 | MAG | 68 | 69.2 | 2.57 | 4.4 | < 0.01 | 0.15 | 47.7 | 88.5 | 0.003 | 0.01 | 5 | 0.74 | 0.13 | 15.1 | 45.62 | < 0.004 | 0.082 | < 0.001 | 22.8 | 93.66 | < 0.001 | 0.07 |
| | NONMAG | 30.2 | 30.8 | 3.28 | 20.8 | < 0.01 | 0.13 | 13.9 | 11.47 | < 0.001 | < 0.01 | 11.4 | 0.3 | 0.048 | 40.5 | 54.38 | < 0.004 | 0.099 | < 0.001 | 3.47 | 6.34 | 0.001 | 0.08 |
| | Calculated Head | - | - | 2.79 | 9.45 | < 0.01 | 0.14 | 37.3 | - | 0.002 | 0.01 | 6.97 | 0.60 | 0.10 | 22.92 | - | < 0.004 | 0.09 | < 0.001 | 16.85 | - | < 0.001 | 0.07 |
| DJ_K 24 2020 | MAG | 34.3 | 49.6 | 2.91 | 7.52 | < 0.01 | 0.12 | 36.4 | 72.23 | 0.003 | 0.01 | 6.79 | 0.58 | 0.14 | 22.1 | 33.5 | 0.007 | 0.25 | 0.002 | 16.3 | 83.7 | < 0.001 | 0.06 |
| | NONMAG | 34.8 | 50.4 | 3.41 | 18.3 | < 0.01 | 0.14 | 13.8 | 27.8 | 0.003 | < 0.01 | 12.3 | 0.28 | 0.046 | 43.3 | 66.5 | < 0.002 | 0.2 | 0.002 | 3.13 | 16.3 | 0.001 | 0.07 |
| | Calculated Head | - | - | 3.16 | 12.95 | < 0.01 | 0.13 | 25.0 | - | 0.003 | < 0.01 | 9.56 | 0.43 | 0.09 | 32.8 | - | 0.003 | 0.22 | 0.002 | 9.7 | - | 0.001 | 0.07 |
| DJ_K 33/A 2020 | MAG | 80.21 | 77.6 | 2.05 | 3.07 | < 0.01 | 0.17 | 53.5 | 92.2 | 0.003 | < 0.01 | 3.7 | 0.79 | 0.097 | 9.71 | 49.24 | < 0.004 | 0.04 | < 0.001 | 26.4 | 95 | < 0.001 | 0.11 |
| | NUNMAG | 23.14 | 22.4 | 3.08 | 22.4 | < 0.01 | 0.12 | 15.7 | 7.81 | 0.003 | 0.01 | 9.82 | 0.28 | 0.055 | 34.7 | 50.8 | < 0.004 | 0.13 | 0.002 | 4.8 | 4.96 | <0.001 | 0.19 |
| DI CD 02 2020 | | - 0 E | 26.0 | 2.28 | 7.40 | < 0.01 | 0.10 | 45.U 26.0 | - | 0.003 | < 0.01 | 5.07 | 0.68 | 0.088 | 15.3 | - | < 0.004 | 0.06 | < 0.001 | 21.56 | - | < 0.001 | 0.13 |
| DJ_GD 02 2020 | NONMAG | 9.0 95.9 | 20.9 73.1 | 3.0 3.7 | 7.05 | < 0.01 | 0.18 | 30.9 147 | 40.00 51.0 | 0.005 | < 0.01 | 0.49 | 0.0 | 0.23 | 43.2 | 13.93 | < 0.004 | 0.11 | < 0.001 | 19.9 | 34.0 | | 0.07 |
| | Colculated Hood | 20.0 | / 3.1 | 3.7 | 19.2 | <0.01 | 0.11 | 20.7 | 51.9 | 0.004 | <0.01 | 10.44 | 0.27 | 0.00 | 43.4 | 04.07 | <0.004 | 0.004 | <0.001 | 3.9 Q J | 34.9 | <0.001 | 0.05 |
| | Galculated Head | - | - | 5.7 | 10.9 | <0.01 | 0.15 | 20.7 | - | 0.004 | <0.01 | 10.44 | 0.30 | 0.12 | 37.5 | - | 0.004 | 0.09 | <0.001 | 0.4 | - | <0.001 | 0.00 |

Debergadé) that originate at the foot of the old Mablas rhyolites and Dahla basalts which are prominent geological formations in the region [3]. As the wadis flow towards the sea [36], they probably carry with them a variety of placer minerals and deposits that are transported from their source rocks (Fig. 1c). The interaction between these geological formations and the hydrological processes in the region contributes to the accumulation and distribution of valuable mineral resources along the coastline. Furthermore, the ancient climate of Djibouti, like much of the Horn of Africa, has undergone significant changes over geological time scales. Although the modern climate is predominantly arid and semi-arid, with hot temperatures and low rainfall, historical and paleoclimate data suggest that the climate was different in various periods, particularly during the Pleistocene to Holocene [38-40]. In fact, successive fluctuations with episodic variations of cooling and wet phases have been described based on higher rainfall and vegetation, identified during the study of the paleolake beds and fossil [39,40]. During this time, the weathering was probably intense along the coastline due to wave action and, combined with the rainfall during wet seasons. This led to the mobilization of sediments containing heavy minerals, which were carried by floods and rivers from inland sources towards the coastline. Once there, these sediments were concentrated along beaches and shorelines by tidal and wave forces. However, it has been suggested that weathering indices based on sediment chemistry do not accurately reflect the integrity weathering history of the source area [41].

At the Godorya site, the samples have a concentration of 8.6 % TiO₂ and 21 % Fe₂O₃. In the Khor Angar area, the values for TiO₂ and Fe₂O₃ range between 12.3 % and 22.1 %. The Obock site exhibits the highest TiO₂ and Fe₂O₃ values ranging from 10.3 % to \sim 24 % and 34.1 % to 53.9 % respectively (Table 1). Trace elements are also present, but they are primary associated with the titanium dioxide. X-ray fluorescence analysis conducted after magnetic separation revealed a titanium concentration of <30 % (Table 1), indicating the presence of titanomagnetite. According to the literature, mining procedures of titanomagnetite are expensive, complex and environmentally polluting [42]. In addition, metallurgical extraction studies of vanadium from titanomagnetite are currently being investigated in countries such as China [42]. Furthermore, in this study ilmenite was detected in the magnetic fraction in the XRD results. Although other minerals such as chromite and magnetite are also present. Ilmenite is the most well-known economic mineral with the highest value in the literature. Numerous studies on heavy mineral sand (HMS) deposits have been reported worldwide [9,10,19,21,22,43]. In East Africa, several studies have highlighted the significance of heavy minerals [9,19,21]. These authors reported 146 Mt of heavy mineral resources, including 2.59 % ilmenite, 0.65 % rutile and 0.29 % zircon. Other studies have shown that the Fort Dauphin mine in Madagascar and the Kwale Kenya mine in Kenya titanium on the elevated beaches. Ali et al. [9] reported that the coast of Somaliland could provide ilmenite as a commercial heavy mineral sand. Titanomagnetite has been observed and studied in a variety of geological settings, including New Zealand, where over 60 % of titanomagnetite is found on the north and west

coasts of the island [44,45], Orokolo Bay in Papua New Guinea, where mining companies are exploring direct extraction of this important critical metal [46], China [47], and Australia [48].

However, in the present study, our samples were taken from the subsurface.

Fig. 5 shows a schematic section of the geology and the location of the black sand in the Obock region. Gulf basalts form the bedrock and fluvial deposits are the most developed sediments in this area. Black sands are more concentrated along the backshore of the beach and less prevalent in the dune. Although the high level of impurities on the Obock may discourage further studies in the lateral extension and depth of the sands, recent studies [42,47,49–51] have shown that the high resource value of vanadium titanomagnetite makes it important to take the risk of developing a method to extract and upgrade titanium, iron and vanadium, even in the presence of high silicate impurities. The only main consideration is that there would be capital costs and increased operating costs associated with extracting and producing vanadium titanomagnetite. The presence of traces of ilmenite, titanomagnetite and chromite in this area indicates the need for future work.

6.2. Novelty and limitations

The presence of traces of ilmenite, titanomagnetite and chromite on the Obock coast is very important and interesting in terms of mineral resources for Djibouti. However, further investigation is needed including geophysical, geochemical and metallurgical studies. The geophysical studies will provide an understanding of the lateral and depth extent of these black sands and helping to estimate the titanium oxide resource. Additionally, further work focusing on the coastal geomorphological environment and sedimentary processes such as the nature and intensity of weathering, distance and duration of transport from the source area to the depositional site, is needed to understand the origin of the black sands. Furthermore, the study of several proxies (REE patterns, grain-sized) combined with tectonic and volcanic activities will be useful in understanding the amount of mineral sand that has been transported from upstream to downstream.

In this study, we collected samples from the entire coastline of Obock up to approximately 20 km from the border. We specifically focused on fifteen samples with higher concentrations of TiO_2 due to the expensive nature of the geochemical analysis. Furthermore, due to the high silica content (30 %–40 %) evidenced by this work, geochemical analyses such as grain mapping should be conducted to determine the quantity of pure and impure grains in a sample and the grain size.

Only these studies will potentially allow us to make far-reaching decisions and then begin a metallurgical study to develop a process for cleaning, extracting and upgrading the titanium in the black sand. This work is costly and may require a significant amount of time to complete.



Fig. 5. Schematically cross-section of the studied area.

7. Conclusion

These studies were undertaken to estimate the presence of heavy mineral concentration and showed relatively low levels of TiO_2 (8.63 % to 23.9 %) and Fe_2O_3 (21 % to 53.9 %). The ZrO_2 content was approximately 0.20 %which could be considered as high value and a high silicate fraction (30 %–40 %). The concentrations of Th, U, La and Ce are mostly at or below their respective detection limits. However, XRD analyses of the magnetic fraction showed the presence of chromite, ilmenite and titanomagnetite. Electron microprobe elemental mapping also showed the presence vanadium (~1 wt. %) in the titanomagnetite. Although titanomagnetite is not widely exploited in the world due to environmental pollution and the cost involved in enriching ores with Fe, Ti or even vanadium, it would be interesting to undertake a project to assess the concentration of titanomagnetite, ilmenite and chromite at depth through drill holes.

Ethical statement

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this study.

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CRediT authorship contribution statement

N. Moussa: Writing – review & editing, Methodology, Investigation, Conceptualization. J. Etoubleau: Writing – review & editing, Supervision, Methodology, Conceptualization. T. Mohamed: Writing – original draft, Methodology. J. Langlade: Writing – original draft, Methodology. M.O. Awaleh: Writing – review & editing, Supervision, Methodology.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this study.

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Supplementary materials

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