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Recovery of valuable heavy minerals via gravity and magnetic separation operations from Diit Quaternary stream sediments, southern coast of the Red Sea, Egypt

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Abstract. This research aims to concentrate and recover valuable heavy minerals (VHMs) contained in Diit area, southern coast of the Red Sea, Egypt. In this study, three technological samples were collected representing Lens, Delta and Wadi Diit; mineralogical investigation followed by mineral processing was performed in order to recover VHM content. VHMs such as zircon, ilmenite, rutile, sphene, apatite, garnet, and magnetite have been recorded in associations with lesser amounts of uranotorite, monazite, xenotime, fergusonite, khatyrkite, and gold. Initially, two stage wet gravity concentration processes, rougher and scavenger, were carried out via a shaking table to produce a clean concentrate of VHMs, which was used as feed material for the magnetic separation process. Magnetic separation was performed in order to separate ferromagnetic minerals from paramagnetic minerals as well as diamagnetic minerals to obtain clean concentration of these fractions. Assay and Material balance of the concentration steps proved that the THM content of Diit lens increased to 89.70% with a recovery of 91.68% in a weight of 61.08% out of the original sample. While the percentage of THM content in Wadi and Delta Diit samples increased to 48.68 and 47.67% respectively, with recovery equivalent to 71.67 and 82.06% respectively in a weight of 16.51, 18.7% respectively out of the original sample. Finally, a process flowsheet was created according to the optimum conditions for concentration and separation processes.

Keywords: Diit Area, VHMs, valuable heavy minerals, wet gravity concentration, Magnetic separation, flowsheet

1. Introduction and geological context

Heavy minerals (HMs) are minerals with higher specific gravity values than quartz (sp.gr. 2.65), as it is the most common rock-forming mineral. The valuable heavy minerals (VHMs) are heavy minerals of economic importance due to their properties and their prevalence.^{1,2} The most valuable and commercially exploitable heavy minerals are; zircon, ilmenite, monazite, rutile, xenotime, kyanite, andalusite, silimanite, staurolite, chromite, garnet, magnetite, columbite-tantalite, cassiterite, wolframite and scheelite as well as gold and platinum. Beneficiation and upgrading of the VHMs in beach sand or in stream sediments are performed firstly using gravity concentration technique; followed by the combination of magnetic and electrostatic separation.³⁻⁶ Flotation can also be used as a later or complementary stage to achieve a cleaner product.⁷⁻⁹ Diit area is located on the southern coast of the Red Sea of Egypt. It is bounded by latitudes of 22° 18' N and 22° 41' N and longitudes of 35° 55' E and 36° 18' E (figure 1) and covered an area about 760 km². Heavy minerals accumulations are observed along the coast of the Red Sea, which were formed not only by transportation processes related to the Red Sea offshore currents, but also by the drainage networks locating in the Eastern Desert of Egypt.¹⁰⁻¹² The rock units of Diit area have a variety of age ranging from Late Proterozoic to Cenozoic rocks. The detailed geological, structural, and petrographical characterization of the study area was previously discussed.¹⁰ The alluvial deposits in the study area cover the area between the Basement Mountains and



the Red Sea coast forming fan shape delta called as "Delta Diit". The Delta Diit has a triangular shape, with its apex trending nearly in the southwest direction at the mouth of Wadi Diit (runs about 85 Km in southeastern desert of Egypt, while its most length about 300 Km extends in Sudan), while its base represents the Red Sea coast. It has a considerable concentration of black sands in the form of lens with about 5km length and width of about 30m extending along the shoreline (figure 1). Diit Quaternary deposits are represented by wadi deposits, sand dunes, sand sheets, and sabkha deposits along the southern coast of the Red Sea (figure 1).

Detailed mineralogical characterization of Diit stream sediments was previously discussed by Fawzy et al., (2022).¹¹ Fawzy (op. cit.) collected and studied ninety four samples that covered the area under investigation (seventy six spot samples in Delta Diit, ten samples in Wadi Diit and eight samples in Diit lens). The mineralogical investigation of this work concluded that the VHM that were recorded are magnetite, ilmenite, garnet, zircon, sphene, apatite, rutile, pyroxene, amphibole, uranothorite, xenotime, monazite, fergusonite, khatyrkite, and gold.

This paper examines the ability for upgrading and concentrating the VHM from Diit Quaternary stream sediments by using the simplest and cheapest physical methods and proposes a flowsheet used as a guide for exploiting the economic heavy minerals presented in the area under investigation and similar ones.

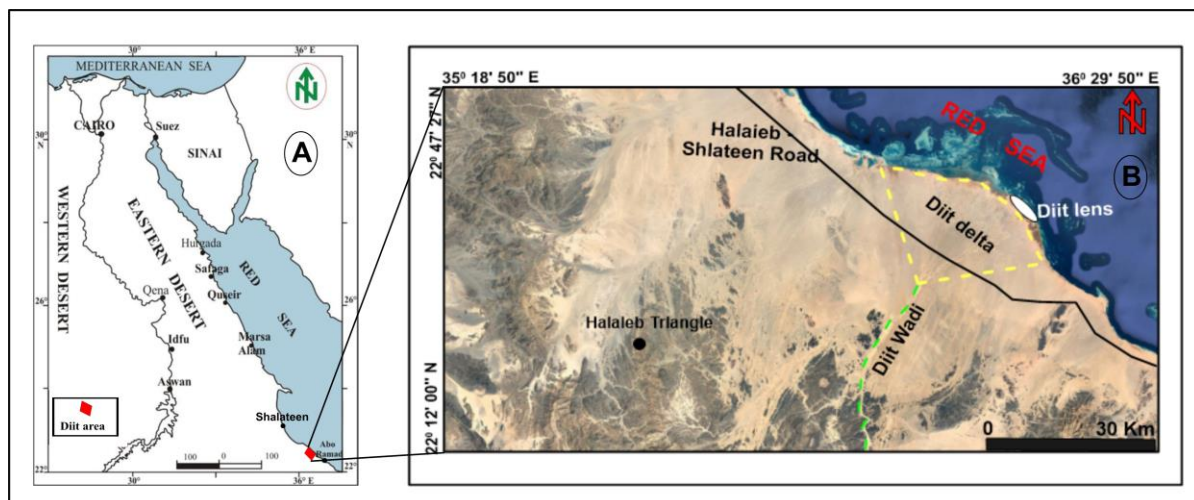


Figure 1. (A) General location map of the study area. (B) Satellite image showing Diit lens, Wadi, and Delta area

2. Sampling and Methodology

Three representative technology samples were used in this work from Wadi, Delta, and Diit lens, each weighing about 20 to 25 kg. Initially, grain size distribution analyses were performed for each of the three samples using a representative sample weighing about 250 g. The samples were sieved using 2, 1, 0.5, 0.25, 0.125 and 0.063 mm (ASTM codes¹³) sieve series representing gravel, very coarse sand (V.C. sand), coarse sand (C. sand), medium sand (M. sand), fine sand (F. sand), very fine sand (V.F. sand) and mud sizes. Heavy-liquid separation tests were carried out on another representative sample weighing about 100 g to determine the THM content using bromoform with a specific gravity of 2.89. Grain size distribution analyses of heavy minerals obtained from bromoform separation were done to determine the size distribution of these target minerals.

The initial mineralogical identification for the heavy bromoform fractions was carried out with the aid of binuclear microscope, and then subjected to scanning electron microscope (SEM) attached with a Philips XL 30 energy-dispersive spectrometer unit (EDS).

The upgradability tests for VHMs were performed via gravity concentration using Wilfley shaking table No. 13¹⁴ for get rid of the light gangue minerals as much as possible. After completion of each concentration stage (rougher and scavenger), a representative sample weighting about 100 g of each of the gravity concentration products (concentrate and tail) is subjected to heavy-liquid separation test using bromoform for determination of the THM assay. The obtained heavy mineral fractions were magnetically separated using Carpco dry high intensity magnetic separator (DHIMS) Model MLH (13) III-5" ¹⁵ to fractionate and separate the ferromagnetic from paramagnetic and diamagnetic minerals and obtain a clean concentrate from these fractions. The magnetic separation processes were achieved at pre-optimized factors of a medium air gap of 1.5 cm, magnetic field current at 1 and 3 amperes, magnetic roll speed 30 rpm and optimum feed rate 39.2 g/min.

X-ray fluorescence analyses were performed for feed samples as well as the heavy mineral concentrates for measuring major and some trace element contents. XRF analyses were carried out via Axios advanced, Sequential WD_XRF Spectrometer.¹⁶ To prepare the samples for XRF measurements, representative samples were ground to a powder (<75 μm) inside an agate mortar and then heated for 2 hours at 1000°C to calculate the total loss of ignition (LOI). For the purpose of mineralogical identification and confirmation, X-ray diffraction (XRD) analyzes was performed on magnetic products using a Bruker D8 Discover X-ray Diffractometer.¹⁷

3. Results and Discussions

3.1. Mineralogical characterization results

As a results of grain size distribution analyses for the studied samples (lens, Delta and Wadi Diit), the percentages of distribution of gravel, very coarse sand, coarse sand, medium sand, fine sand, very fine sand and mud are presented in (figure 2). The samples show a high degree of homogeneity: indeed the sandy fraction, with about 97.98, 90.85 and 97.32% of total percentages in weight of Diit lens, Delta and Wadi samples respectively on average, was most present, while the others, gravelly and silty-clayey fractions, showed lower percentages in weight. Cumulative percent passing curves for the representative samples of Diit lens, Delta and Wadi were presented in (figure 3). From these curves, the effective diameter values of D_{80} are 0.32, 0.6, and 0.36 for Diit lens, Delta and Wadi respectively.

For calculating the VHM content for each sample of Diit area, about 100 representative grams were separated using bromoform (CHBr_3 , sp. gr. of 2.89) and the ratio of heavy economic mineral to gangue light silicate minerals were calculated. The light fractions of Wadi and Delta Diit samples represent higher values (80.87% mass for Delta Diit sample and 79.49% mass for Wadi Diit sample) while the lower value represents the lens sample (39.15%) which is relative to the presence of high values heavy mineral content. Microscopic investigation of the light gangue fractions proved that they contain mainly of quartz, feldspars and micas

The content of the heavy fraction in the Diit lens sample (59% mass) is greater than those of Wadi and Delta Diit sample (11.45% and 12.48% mass respectively). The microscopic examinations of heavy fractions with the help of SEM analyses proved that ilmenite, magnetite, zircon, sphene, apatite, garnet, rutile and heavy green silicates (pyroxene, epidot, and amphibole) are the constituent minerals of all Diit area samples, but in varying proportions between Wadi, Delta and lens. Also present, in minor or trace amounts, are uranothorite, xenotime, monazite, fergusonite, and khatyrkite (figures 4 and 5). As shown in (figure 2), grain size distribution analysis of the heavy fractions indicates that most of the heavy minerals are present in the sandy fraction (99% Diit lens, 93% Delta Diit and 90.5% Wadi Diit). The remaining percentage of the heavy minerals is present in the mud fraction, while the gravel fraction in all samples does not contain heavy minerals.

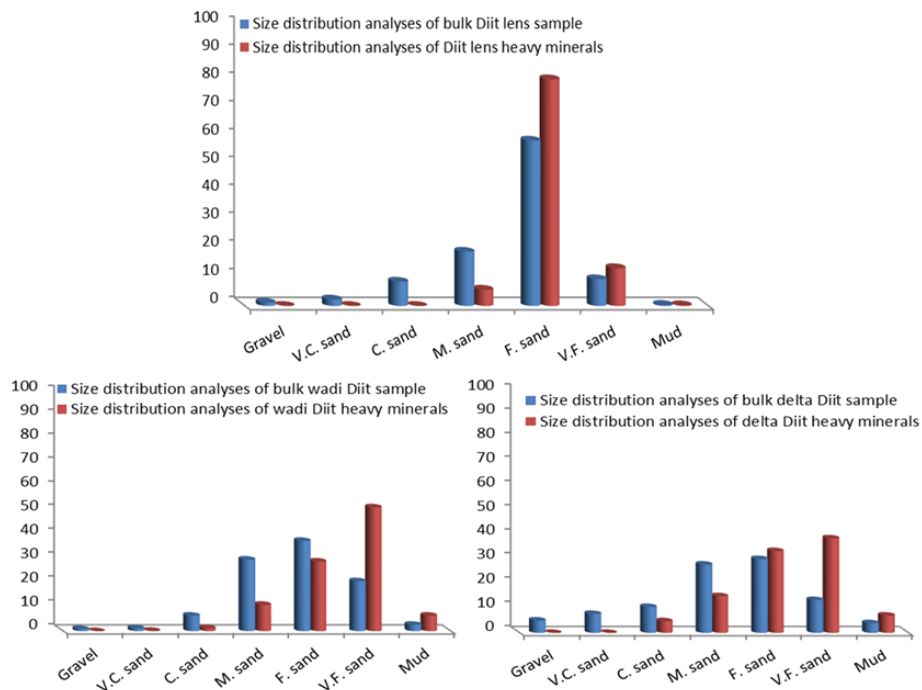


Figure 2. Histogram showing the grain size distribution analyses for Diit samples (lens, Wadi, and Delta) in comparison with their heavy mineral distribution analyses.

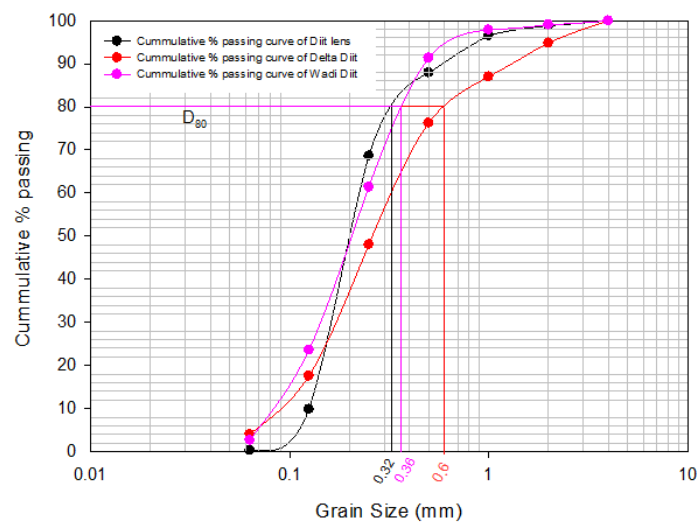


Figure 3. Grain size distribution analyses of Diit lens, Wadi and Delta samples.

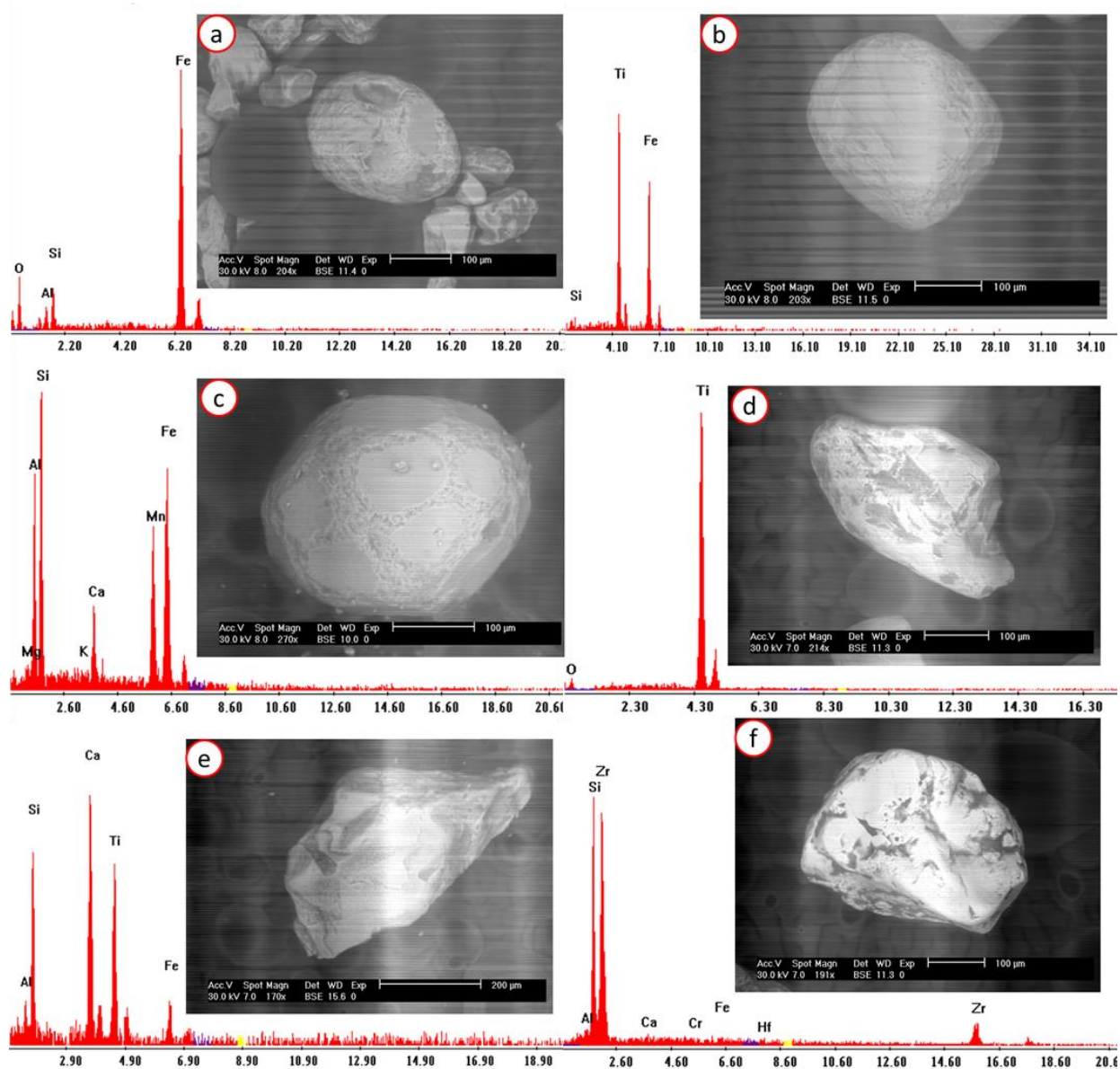


Figure 4. Back-scattered electron (BSE) images and corresponding EDS spectra for magnetite (a), ilmenite (b), garnet (c), rutile (d), sphene (e), and zircon (f)

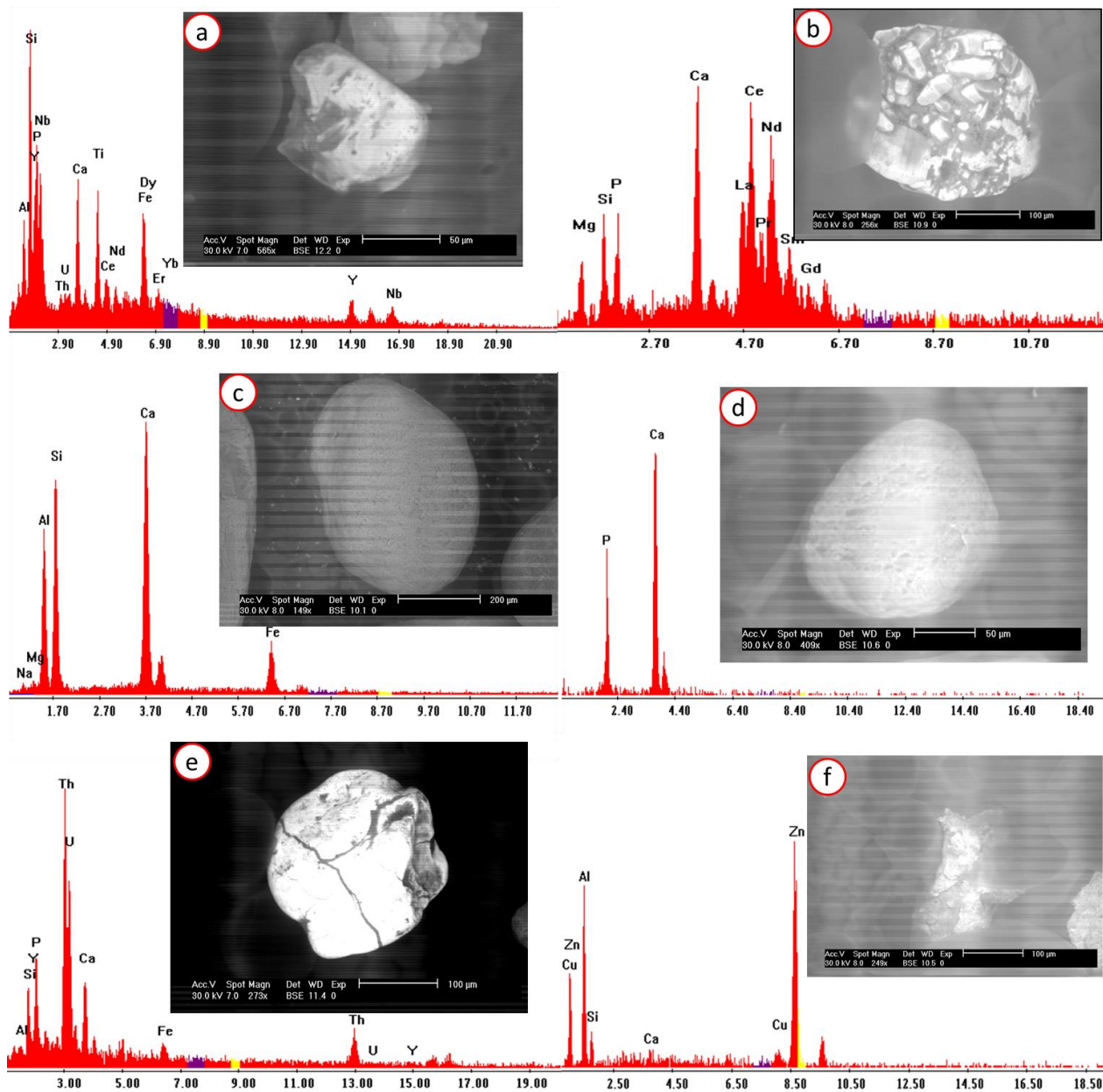


Figure 5. Back-scattered electron (BSE) images and corresponding EDS spectra for fergusonite (a), xenotime (b), green silicates (c), apatite (d), uranothorite (e), and khatyrkite (f)

3.2. Feasibility of physical upgrading

Usually, the physical upgrading processes of the economic heavy minerals found in black sand or stream sediments start with gravity concentration processes in order to eliminate the low density gangue silicate minerals such as quartz and feldspar, which are present in large proportions and produce a heavy mineral concentrate (HMC). Then followed by the stage of magnetic separation of the obtained HMC, in order to separate ferromagnetic from paramagnetic and from diamagnetic heavy minerals.¹⁸⁻²⁰

It is necessary to evaluate the gravity concentration process in terms of whether it is possible or not, so the concentration criterion (CC) is commonly used for this purpose. A concentration criterion (CC) can be defined as;

$$\text{Concentration Criterion (CC)} = \frac{\text{Sp. gr. of heavy mineral} - \text{sp. gr. of fluid medium}}{\text{Sp. gr. of light mineral} - \text{sp. gr. of fluid medium}}$$

The separation efficiency decreases with decreasing the criterion value, with values > 2.5, possible separation occurs.^{21, 22} Concentration criterion values for the heavy minerals in the Diit sample relative to the main gangue minerals (quartz and feldspar) are tabulated in Table 1. The data shows that gravity separation of some VHMs (magnetite, ilmenite, zircon, rutile, uranotorite, monazite, xenotime, fergusonite, khatyrkite, and gold) from the silicates gangue minerals will be possible, whereas separation of other heavy minerals as apatite, sphene, garnet, amphibole, epidote and pyroxene, from the gangue silicate minerals will be relatively difficult. So, several rounds of scavenging concentration steps were carried out after the rougher step to recover the target minerals that were not recovered during the initial roughing stage. The scavenging concentration stages were achieved by changing the concentration conditions (water flow, feed rate, deck tilt, and amplitude) to make them more rigorous than the initial roughing.

3.2.1. Shaking table concentration. In order to eliminate the high percentages of light gangue minerals (quartz and feldspar) in Diit area samples (80.87%, 79.49%, and 39.15% mass for Delta, Wadi, and lens of Diit sample respectively), wilfely shaking table no. 13 was used to obtain a clean concentrate of the VHMs. To obtain higher recovery values, a rougher concentration step was initially performed, and then the obtained tail was subjected to scavenging concentration step to recover the remaining heavy minerals that were not recovered during the initial roughing stage. Two rounds of the scavenging concentration stages were carried out on the Wadi and Delta Diit samples, while the Diit lens sample required only one round for scavenging operations.

The operation conditions for the roughing and scavenging concentration stages using shaking table as feed rate, water flow rate, stroke length and inclination angle were optimized and listed at (figures 7 and 8). It is quite clear that the scavenging stage has greater values of the operation conditions than the roughing stage, and this comes as a result of the increase in the proportion of light gangue minerals and the decrease in the heavy minerals during the scavenging stages compared to the roughing stages.

The final concentrates obtained after several wet gravity concentration operations for the Wadi, Delta and Diit lens samples were collected, dried, weighed, and analyzed elementally using XRF spectrometry and compared with XRF analyses of the feed samples and presented in (table 2) in addition to the calculated elemental improving ratio (EI). Improving ratio of elementals is calculated by dividing the grade of concentrate by the grade of the feed (c/f) and its value refer to how many times the concentrate has element concentration relative to the feed.

From the results of (table 2), the values of the improving ratio for the major elements SiO₂, Al₂O₃, K₂O, and SO₃ indicate that their grades were reduced in the concentrate than in the feed, and this indicates a reduction in the percentages of the gangue silicate minerals that have low specific gravity

(quartz and feldspar) as a result of wet gravity separation processes via shaking table. On the contrary, the improving ratio values for the elements Ti, Ni, Fe, Zn, Mn, Pb, Y, Zr, Cr, and Nb indicate a doubling of their grade in the concentrate than in the feed sample, and this related to a doubling of the ratios of their minerals such as ilmenite, magnetite, rutile, sphene, zircon, and garnet in the concentrate as a result of the high value of its specific gravity during wet gravity separation processes.

Table 1. Concentration criteria for separation of heavy minerals from gangue minerals.

Heavy Minerals		Gangue Minerals	
Mineral	Sp. gr.	Quartz (sp. gr. 2.62)	Feldspar (sp. gr. 2.56)
Magnetite	5.19	2.59	2.69
Ilmenite	4.79	2.34	2.43
Zircon	4.6	2.22	2.31
Apatite	3.2	1.36	1.41
Sphene	3.5	1.54	1.60
Rutile	4.20	1.98	2.05
Garnet	3.50	1.54	1.60
Pyroxene	3.50	1.54	1.60
Amphibole	3.2	1.36	1.41
Epidote	3.4	1.48	1.54
Uranothorite	5.5	2.78	2.88
Monazite	5.26	2.63	2.73
Xenotime	4.75	2.31	2.40
Fergusonite	5.7	2.90	3.01
Khatyrkite	4.35	2.07	2.15
Gold	17	9.88	10.26

Assay and material balance of the wet gravity concentration processes for Wadi and Delta Diit samples after roughing and two rounds of scavenging operations were presented in Figure 7. The results revealed that the THM content was raised from 11.45 to 48.68 % for Wadi Diit sample and from 12.48% to 47.67% for Delta Diit sample in 16.51 and 18. 7% by mass yield for Wadi and Delta Diit samples respectively. It means that 71.67 and 82.06% of the THM content was saved in the final concentrate for Wadi and Delta Diit samples respectively and 28.33 and 17.94% of the THM content was lost at the tailing of the Wadi and Delta Diit samples respectively. The improving ratio of the Wadi and Delta Diit samples are 4.25 and 3.92% respectively, this means that the Wadi and Delta Diit concentrate have 4.25 and 3.92 times respectively the THM concentration of the feed.

Figure 8 displays the material balance of the tabling concentration processes for Diit lens sample after roughing and one round of scavenging operation. The material balance results revealed that the THM content was raised from 61.89 to 89.70% in 61.08 % by mass yield; it means that 91.68% of the THM content was saved in the final concentrate and 8.32% of the THM was lost at the tailing. The value of the Diit lens improving ratio means that the concentrate has 1.5 times the THM concentration of the feed.

Table 2. XRF elemental analyses demonstrating the feed grade, concentrate grade and improving ratio (ER) for Diit samples (lens, Wadi and Delta).

Sample	Diit Lens			Delta Diit			Wadi Diit		
	Feed grade (f)	Conc. grade (c)	IR (c/f)	Feed grade (f)	Conc. grade (c)	IR (c/f)	Feed grade (f)	Conc. grade (c)	IR (c/f)
Major elemental oxide in wt. %									
SiO ₂	29.60	23.61	0.80	59.84	51.39	0.86	59.88	45.95	0.77
TiO ₂	9.04	20.11	2.22	0.94	2.34	2.49	0.89	1.91	2.15
Al ₂ O ₃	7.41	7.72	1.04	14.37	13.23	0.92	14.81	13.30	0.90
Fe ₂ O ₃ ^{Total}	23.78	33.15	1.39	8.26	14.14	1.71	8.71	11.53	1.32
MnO	0.904	1.40	1.54	0.163	0.325	1.99	0.164	0.260	1.59
MgO	3.85	3.99	1.04	3.14	5.09	1.62	3.00	4.66	1.55
CaO	18.06	6.88	0.38	5.75	7.36	1.28	5.14	6.91	1.34
Na ₂ O	0.87	1.03	1.18	2.47	2.93	1.19	2.26	3.17	1.40
SO ₃	0.12	0.11	0.92	0.13	0.03	0.23	0.08	0.02	0.25
K ₂ O	0.36	0.23	0.64	2.05	0.69	0.34	2.03	0.74	0.36
P ₂ O ₅	0.25	0.30	1.20	0.30	0.32	1.07	0.33	0.32	0.97
L.O.I.	4.57	0.26		2.31	1.76		2.43	1.86	
Trace elements in ppm									
Ni	----	88.8	88.8	133.58	239.9	1.80	110.01	195.82	1.78
Zn	208.88	549.9	2.63	104.44	494.7	4.74	128.54	583.57	4.45
Cu	87.88	25.7	0.29	95.86	48.4	0.50	119.83	65.91	0.55
Pb	-----	15.7	15.7	46.42	477.0	10.28	-----	11.3	11.3
Sr	1818.04	841.3	0.46	752.58	1748.4	2.32	693.39	1698.81	2.45
Rb	54.87	9.2	0.17	100.59	51.2	0.51	91.44	41.15	0.45
Zr	3109.26	6982.9	2.25	555.23	3878.2	6.98	421.97	2958.01	7.01
Cr	2360.66	7605.3	3.22	403.71	2136.0	5.29	451.61	2262.57	5.01
Ga	37.20	29.6	0.80	22.32	99.2	4.44	44.64	188.83	4.23
Y	133.86	247.9	1.85	47.25	254.1	5.38	31.50	173.57	5.51
Co	432.58	67.5	0.16	-----	114.9	114.9	-----	100.9	100.90
Nb	167.77	309.0	1.84	-----	120.4	120.4	-----	105.44	105.44

3.2.2. Magnetic separation results. Magnetic separation processes via DHIMS produced four magnetic fractions, which are as follows; Ferromagnetic, strongly paramagnetic, weakly paramagnetic and finally the diamagnetic mineral fraction. Ferromagnetic fraction percentages are 1.56%, 2.19% and 3.16% mass for Diit lens, Wadi and Delta samples respectively while the strongly paramagnetic fraction at 1 ampere reaches 38.05% for the lens, and reaches 25.81 and 7.62% for the Wadi and Delta Diit samples respectively. As for the results of separating the weakly paramagnetic fraction at 3 amperes, they are

20.33, 28, and 44% for the lens, Wadi and Delta samples respectively. Finally, the diamagnetic fraction has percentage of 40.07 for lens sample and 44, 44.69 for Wadi and Delta Diit sample respectively.

X-ray diffraction analyses are used to identify and confirm the mineral contents of the different magnetic fractionations and presented in (figure 6). The ferromagnetic fraction diffraction lines are in accordance with COD card no. 9005812 for magnetite, COD card no. 9015065 for hematite, and COD card no. 9002332 for magnetite-h (figure. 6a). Pure concentrate of ilmenite occurred and confirmed at the paramagnetic fraction that has been separated at 1ampere field current and has diffraction lines in accordance with COD card no. 1011033 for ilmenite (figure. 6b). The paramagnetic fraction that has been separated at magnetic field current 3 amperes has diffraction lines in accordance with large group of paramagnetic minerals as follows; COD card no. 9000299 for epidote, COD card no. 9001647 for monazite, COD card no. 9001409 for garnet, COD card no. 1008932 for xenotime, COD card no. 9011839 for fergusonite, COD card no. 9000597 for pyroxene, and COD card no. 9001548 for amphibole (figure 6c). Whereas the non-magnetic fraction that has been separated at 3 amperes magnetic fraction has diffraction lines in accordance with COD card no. 1011263 for zircon, COD card no. 9010050 for apatite, COD card no. 9001681 for rutile, COD card no. 9000820 for uranophane, COD card no. 9008463 for gold, and COD card no. 9012196 for khatyrkite (figure. 6d).

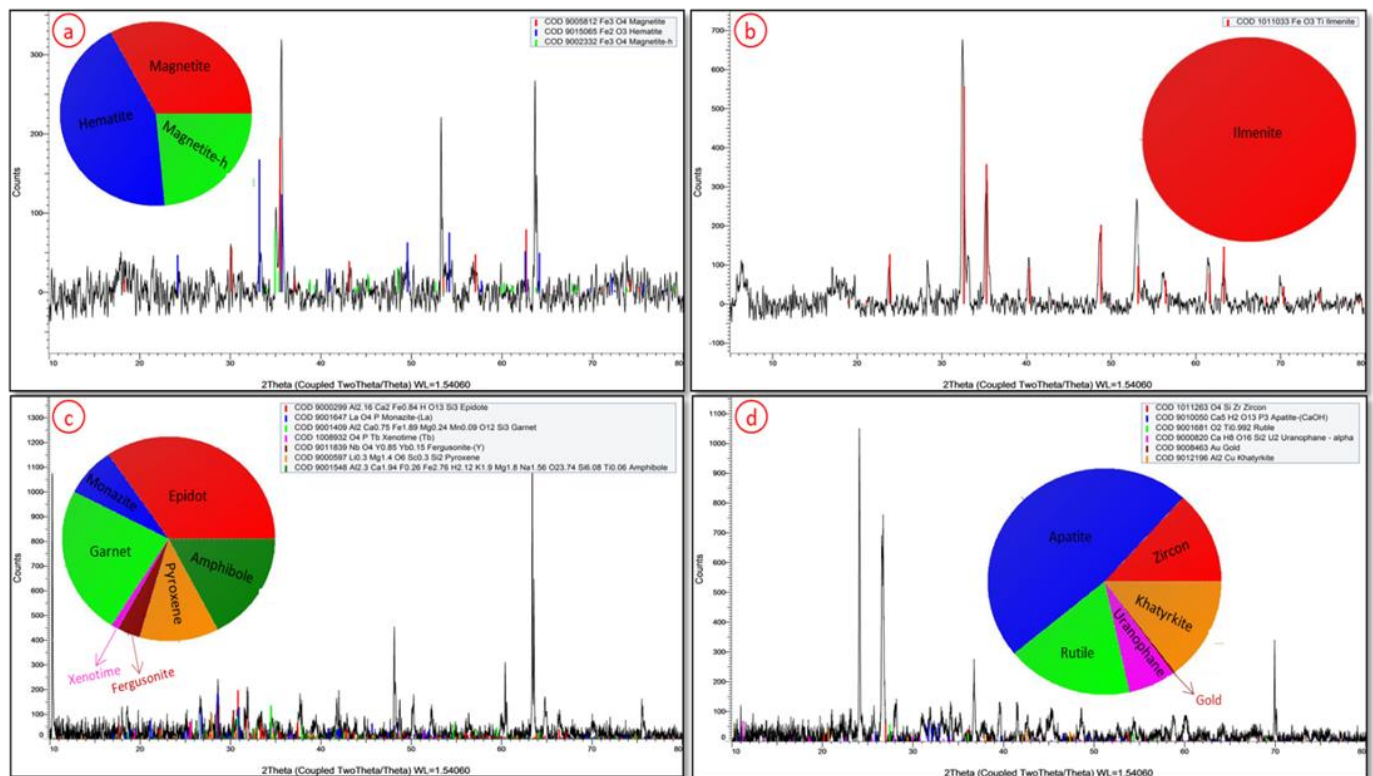


Figure 6. XRD diffractograms showing: a. Mineral content of the ferromagnetic fraction, b. Mineral content of the paramagnetic fraction at 1 ampere, c. Mineral content of the paramagnetic fraction at 3 amperes, and d. Mineral content of the diamagnetic fraction at 3 amperes.

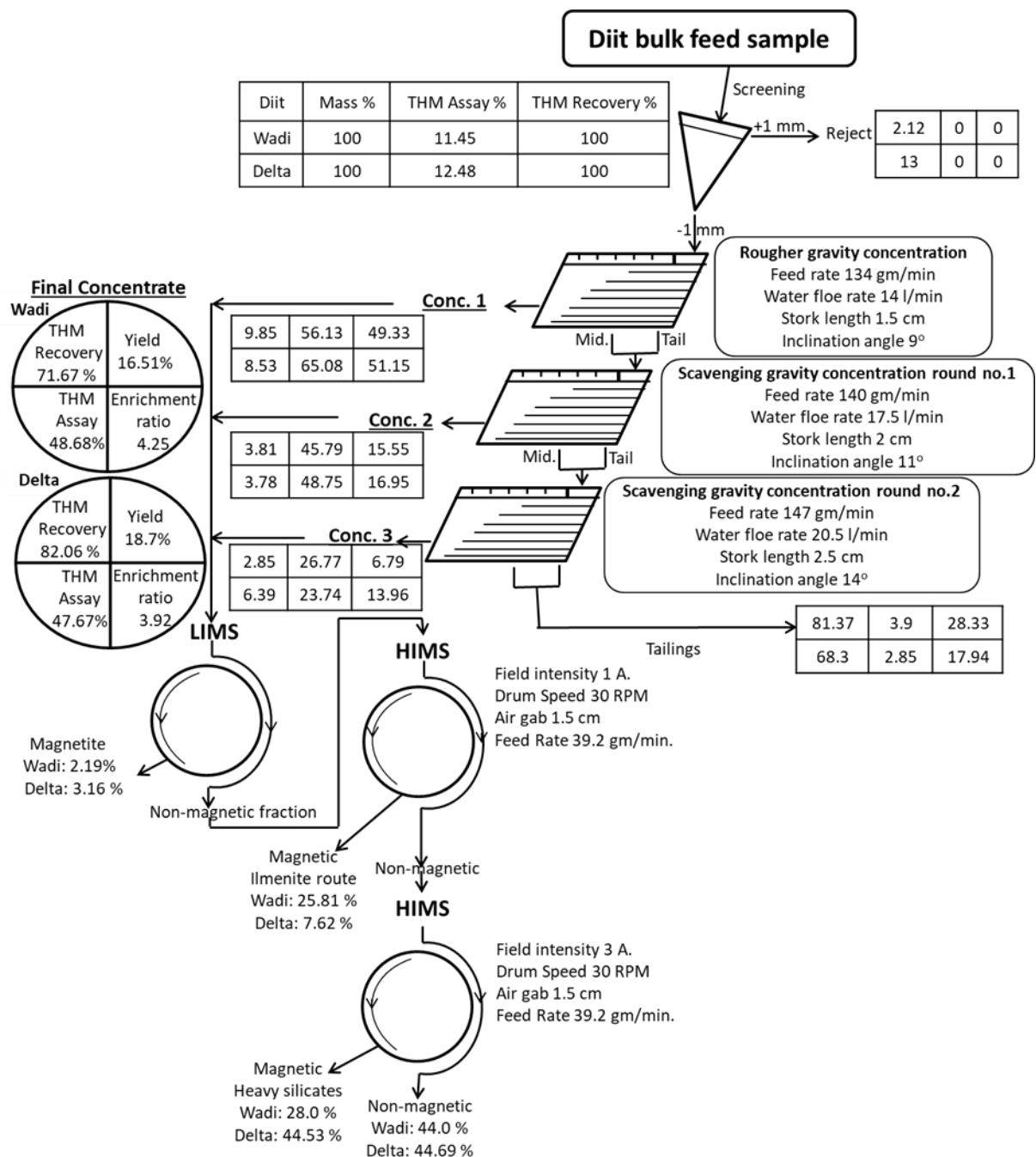


Figure 7. Flowsheet with material balance for recovery of THM from Wadi and Delta Diit feed sample under optimum conditions.

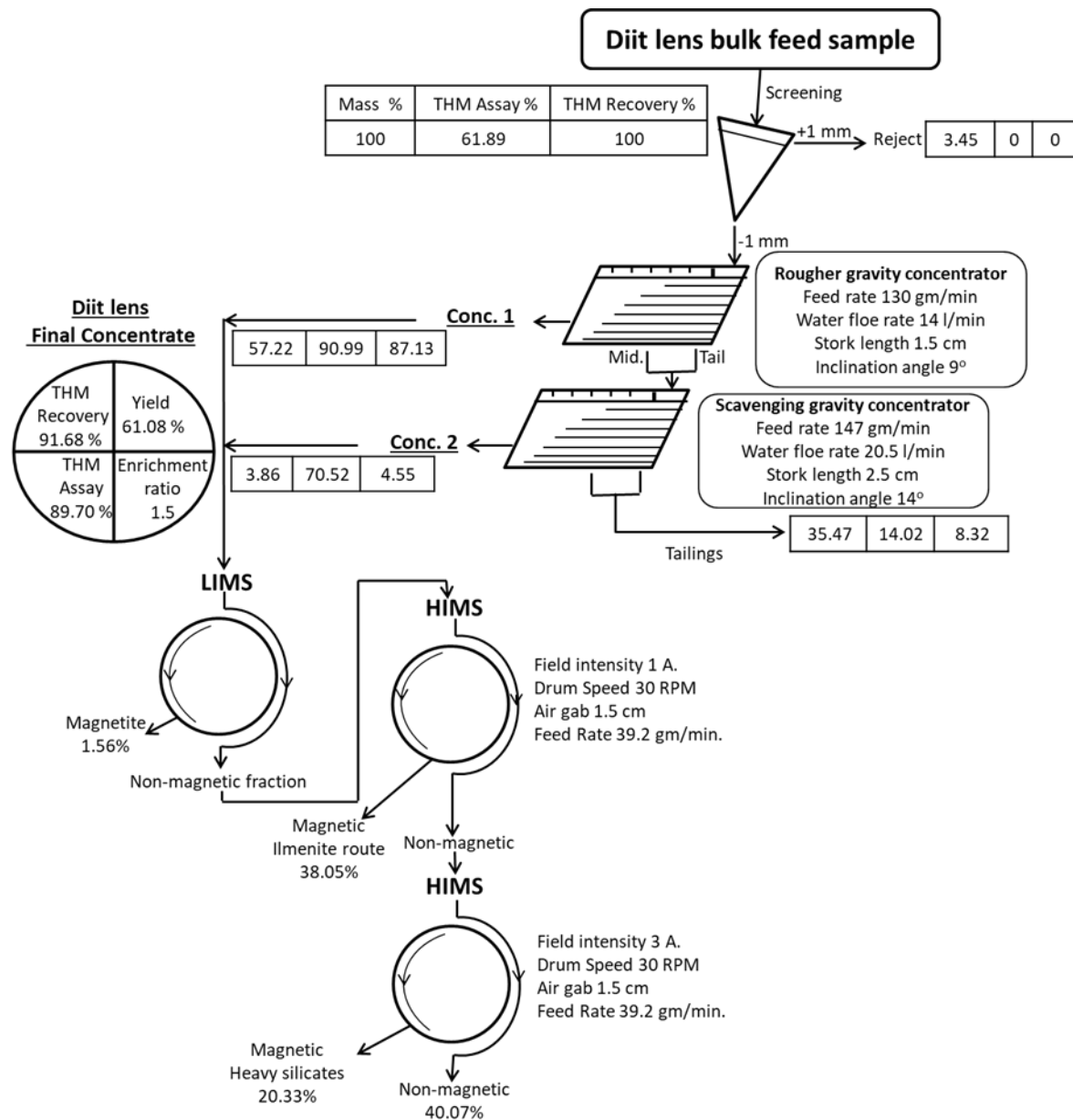


Figure 8. Flowsheet with material balance for recovery of THM from Diit lens feed sample under optimum conditions.

4. Conclusions

Recovery of VHMs from Diit stream sediments was successfully performed in this study using wet gravity concentration tool in conjunction with magnetic separation tool (Carpco DHIMS). Gravity concentration operation tests resulted that the content of SiO_2 , Al_2O_3 , K_2O , and SO_3 is significantly degraded in the concentrate relative to the feed samples while on the contrary, the contents of Ti, Fe, Mn, Ni, Zn, Pb, Zr, Cr, Y, and Nb were upgraded several times in the concentrate as compared to the feed sample. This means that the contents of the VHMs of ilmenite, magnetite, rutile, sphene, zircon, and garnet were increased in the concentrate while the contents of gangue silicate minerals (quartz and feldspar) decreased in the concentrate and increased in the tail.

Assay and material balance of the scavenging after roughing step of the Diit lens sample revealed that the THM assay was raised from 61.89 to 89.70% in 61.08 % by mass yield with of recovery is 91.68%. The THM assay in the Wadi and Delta Diit samples increased from 11.45 to 48.68 and from 12.48 to 47.67% respectively after two rounds of the scavenging concentration step following the roughing step. The THM recovery for the Wadi and Delta Diit samples was 71.67 and 82.06% respectively in a weight of 16.51, 18.7% respectively out of the feed sample with THM loss 28.33 and 17.94 % respectively for Wadi and Delta Diit at the end of the whole process.

DHIMS succeeded in separating VHMs into four fractions; the first fraction is the magnetite concentrate, the second fraction is the ilmenite concentrate, and the third fraction is a concentrate of the green heavy paramagnetic silicates minerals as pyroxene, amphibole, garnet and epidot as well as trace amounts of monazite, xenotime and fergusonite. Finally, the fourth fraction is a concentrate for the diamagnetic minerals as zircon, rutile, apatite, sphene, gold, and khatyrkyite.

Declaration

Conflict Of Interest

The authors declare that they have no conflict of interest.

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