Recovery of Cassiterite and Topaz Minerals from an Old Metallurgical Dump, Eastern Desert of Egypt

Suzan Sami Ibrahim (✉ suzansibrahim@gmail.com)

Central Metallurgical Research and Development Institute <https://orcid.org/0000-0001-7486-2306>

Ayman Hagrass

Tebbin Institute for Metallurgical Studies

khaled Yassin

Central Metallurgical Research and Development Institute

Wael Fathy

Al-Azhar University

Tawfik Boulos

Central Metallurgical Research and Development Institute

Research Article

Keywords: Cassiterite. attrition milling. shaking table. RER dry high intensity magnetic separation

Posted Date: September 8th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-367732/v1>

License: This work is licensed under a Creative Commons Attribution 4.0 International License.

[Read Full License](#)

Version of Record: A version of this preprint was published at Journal of Minerals and Materials Characterization and Engineering on January 1st, 2022. See the published version at <https://doi.org/10.4236/jmmce.2022.101005>.

Abstract

Huge amounts of tailing dumps as a result of mines' blasting operations were impacting both economical and environmental problems. This issue was in serious need to be treated with suitable solutions. Evaluation of one of these tailing dumps in the Eastern Desert of Egypt showed the presence of reasonable amount of cassiterite mineral reaching 0.199%. The mineral was found as finely disseminated particulates within varieties of quartz-feldspar-hornblende-biotite granitic formations.

In the present study, the processing regime relied upon the synergy between reaching liberation size, and mineral over grinding due to its extreme brittleness. However, delicate grinding via attrition scrubbing was adopted to produce $-0.51 + 0.074$ mm attrition product with fine fractions, reaching 62.31% and 37.59%, respectively.

The recovery of cassiterite from the $-0.50 + 0.074$ mm size fraction was accomplished by the physical difference between mother granitic formations that shielded the mineral grains. Under these conditions, joint shaking table/dry high intensity magnetic separation techniques were conducted to recover cassiterite mineral. The CCD statistical system was used as a mathematical approach to optimize the effect of the main working parameters of the magnetic separator, i.e., splitter inclination angle, and belt speed, and their interactions on the cassiterite recovery of the final concentrate. The suggested flow sheet succeeded to recover cassiterite mineral with a grade reaching 11.25% SnO_2 with 94.08% operational recovery from a feed contained 0.19% SnO_2 . These results are highly imperative to achieve applicable processing flow-sheet of such kind of minerals' secondary resources.

Introduction

The Egyptian Eastern Desert contains very large quantities of mine blasting remnants which are accumulated after different mining operations to extract valuable minerals. In fact, many of the scientific studies have succeeded to a large extent in developing research plans to concentrate many important minerals from these wastes such as gold, silver, copper, lead and zinc, and other important ores. However, very few studies have been devoted to recover the mineral cassiterite Fig. 1. In Egypt, tin deposits are classified as mineralization related to plutonic rocks by either as albite-muscovite granites of calc-alkaline to peralkaline affinity having disseminated Ta–Nb–Sn in Abu Dabbab area, as pegmatites related to late granites having Sn, Be, Mo, U, Th, Nb, REE mineralization, or as quartz-carbonate veins related to granite cupolas having Au, Sn, W, Mo, Be and F in Igla area (El Aref et al. 2020).

Cassiterite is considered to be the only economically significant tin mineral in the earth's crust. The mineral is hard and heavy but also extremely brittle. Cassiterite has the chemical formula SnO_2 (78.8 % Sn, and 21.2 % O_2), density of 7.02 g/cm^3 , and Mohs hardness 6–7. Cassiterite is of black, brown-black, yellowish brown, reddish brown coloration. It is nonmagnetic and conductive for electricity (Yang et al. 2014; Angadi et al. 2015; Kumar et al. 2019).

Cassiterite is mainly enriched in the “tin granites,” which contain approximately 15–50 g Sn/ton. Cassiterite also occurs as “mine tin” in domes of granite intrusions, in feldspar topaz granites, and titanomagnetite deposits. Because cassiterite is very resistant to all weathering influences, it is a typical placer mineral. There are no specifications for commercial concentrates of cassiterite because cassiterite is always smelted directly, depending on the concentration, without further treatment (Nandi et al. 2019; Dalimunthe et al. 2020).

Unfortunately, the relative high hardness is accompanied by the unfortunate quality of extreme brittleness. This factor must be taken into account during the crushing and grinding operations prior to concentration (Yang et al. 2019). This is because significant quantities of very fine particles are produced at these steps, resulting in losses of tin in the succeeding processing stages. Therefore, the general policy is that cassiterite grains should, where possible, be recovered at the earliest possible stage and at their largest size to avoid softness that is difficult to handle (Falcon 1982).

It is obvious from the physical characteristics of cassiterite that its high relative specific gravity in relation to its accompanied gangue constituents makes it an ideal mineral for the application of gravity-separation techniques. Development of cassiterite beneficiation techniques has been carried out using flotation, electro and bio-flotation (Sreenivas and Padmanabhan 2002; Pecina et al. 2009; Bulatovic 2010; Gonzales et al. 2013; Gerald et al. 2013; Zhou et al. 2014; Yang et al. 2014; Tian et al. 2017a; Tian et al. 2017b; Tian et al. 2018a; Tian et al. 2018b; Tian et al. 2018c; Tian et al. 2018e; Tian et al. 2019; Zhang et al. 2021). Cassiterite was separated using magnetic separation (Wills and Napier-Munn 2006; Lungu 2009; Jiao et al. 2011; Li and Yang 2016; Zeng et al. 2015; Zong et al. 2018; Slusarek and Zakrzewski 2012; Zeng et al. 2015; Hisayoshi et al. 2016; Dimova et al. 2019; Shvedchikova et al. 2020). Hydrometallurgical separation, and microwave pre-treatment were adapted to recover cassiterite mineral and tin metal (Nakahiro and Wakamatsu 1993; Egbe et al. 2013; Ajaka et al. 2014; Mouedhen et al. 2018; Yang 2014; Saisinchai et al. 2016; Zhang et al. 2021).

Figure 1. Occurrences of cassiterite deposits in the Eastern Desert of Egypt (EGSMA 1998)

Experimental

A technological sample from the tailing dumps of the Eastern Desert of Egypt was supplied for evaluation and treatment. The sample was about – 6 mm in size. It was subjected to elemental evaluation using XRF unit model PANalytical-Axios, and Perkin-Elmer Analyst 200 atomic absorption. The phase analysis of the sample was applied using X-ray diffract-meter model “pw 1010” with CuK α radiation under target voltage 40 kV and current 30 mA in a scanning rate of 5° 2 θ /min. Scanning electron microscope (SEM) of the type JEM-1230, JEOL equipped with energy dispersive X-ray (EDX) spectrometers was used to evaluate the final products.

A representative sample at the size fraction – 6.0 + 2.28 mm from the original sample (about 14% by wt. of the original sample) was divided into different samples cuts according to grain color. Polished sections of these samples were prepared for reflected light microscope investigation.

Size reduction of the original sample was conducted via wet grinding attrition technique using Union Process “Attritor” model 1S. The attrition time was conducted for 15 min as three steps, in closed circuit with 0.50 mm vibrating screen. The impeller speed was 1500 rpm, and the solid/water/balls ratio was 1/1/1 by wt. (each was 800 g). The grinding zirconium ball diameter was 2 mm. The ground product was further screened on 0.074 mm sieve. Both the over and under screens products were collected, dried, and directed to evaluation. The under screen – 0.074 mm fines was kept aside for separate study. The over screen product was further divided into – 0.5 + 0.211 mm and – 0.211 + 0.074 mm fractions for gravity and magnetic separation treatment, separately.

The sample under study could be grouped according to density into light minerals and heavy minerals. The lights included albite, quartz, clinochlore, and talc with average density 2.65 g/cc. On the other side, heavy minerals included hornblende, tremolite, and biotite with average density 3.50 g/cc. The difference in density in this case was about 1.52 which was suitable for efficient table separation of this sand sized sample – 0.51 + 0.074 mm. However, the presence of included heavy cassiterite grains (density reaching 8 g/cc) in addition to other heavy metals like iron, zinc, and copper that were imbedded in the structure of the heavy granites may increase the probability for efficient separation by gravity techniques.

The size fractions – 0.51 + 0.211 mm and – 0.211 + 0.074 mm were subjected separately to a “Wilfley” shaking table. Throughout the experiments, the sample was kept in suspension at 20–25% of solids by weight by mechanical stirring. Increasing of slurry concentration more than 25% led to over-crowding of the particles in the course of separation which deteriorate the separation efficiency. However, the slurry was fed onto an inclined plate at feeding rate of 1.30 kg/min. The wash water flow rate was kept at 20 l/min with table speed in the vicinity of 300 rpm. Stroke length and deck inclination were optimized during the separation study.

The middling products after the separation of the two fractions – 0.51 + 0.211 mm and – 0.211 + 0.074 mm were further subjected to tabling at relatively lesser wash water flow rate, lesser tilt, and a shorter stroke length than the primary separation working conditions. An increase in the wash water flow rate shows increase in the grade of the concentrate fraction at both lower and higher feed flow rates. As wash water flow rate increases, the transport of gangue minerals to the tailings fraction increases which in turn improves the grade of the concentrate fraction. Higher grade of the concentrate fraction is obtained at higher level of both deck tilt angle and feed flow rate. With a lower level of deck tilt angle, an increase in the feed rate decreases the quality of the concentrate fraction (Wills and Napier-Munn 2006; Tripathy et al. 2010; Egbe et al. 2013; Ajaka et al. 2014; Mouedhen et al. 2018).

At the end of shaking table process, various products were collected, dried, and directed to evaluation. The well dried products were fed separately onto laboratory Eriez RER dry high intensity magnetic separator at permanent field strength of 21,000 gauss. Feeding rate was adjusted at 2 kg/ hr. Box-Behnken Design (BBD) was constructed to determine effect of the separator splitter inclination and belt speed and their interactions on the cassiterite recovery% as the sole response of this design, and to identify its optimum conditions as well, Table 1.

The 2^k factorial design, which is basically a series of tests containing k factors, each of which has two levels ('low' and 'high'). The target a factorial experiment is mainly to determine the factor effects on the process output. Understanding factor effects helps to adjustment of these factors to optimize the process output. The effect of each factor on the process output can be due to single factor effect and/or its interaction with one or more from the other factors (interactive effects). The magnitude and sign of the numerical values of different effects coefficients give good idea about how these effects influence the process output. A higher value for an effect coefficient means that the factor affects the process output significantly. A positive and negative signs means that increasing the level(s) of the factor(s) will increase or decrease the output of the process respectively.

Software package, Design-Expert 6.0.5, Stat-Ease, Inc., Minneapolis, USA, was used for regression analysis of experimental data and to plot the response surface (Box and Behnken 1960). Analysis of variance (ANOVA) was used to estimate the statistical parameters. The extent of fitting the experimental results to the polynomial model equation was expressed by the determination coefficient, R². F-test was used to estimate the significance of all terms in the polynomial equation within 95% confidence interval. The products after magnetic separation were directed to evaluation.

Table 1

Levels of the two variable parameters

Variable	Symbol	Unit	(-)	(0)	(+)
Splitter angle	A	degree	70.00	72.50	75.00
Belt speed	B	rpm	100	150	200

Results And Discussion

Sample characterization

Results of XRF analysis showed that SnO₂ constituted 0.199% of the sample. The major oxides were SiO₂, Al₂O₃, MgO, Fe₂O₃, and CaO. The oxides contents assayed 51.94%, 11.87%, 10.97%, 8.73%, and 5.68%, respectively, Table 2. They were related to different minerals phases, e.g. albite, quartz, tremolite, clinocllore hornblende, biotite, and talc, Fig. 2. According to XRD pattern, semi quantitative analysis, these phases represented about 27.30%, 22.8%, 12.30%, 21.90%, 11.70%, and 4.00%, respectively, of the original sample, Fig. 2. Rare earth oxides e.g. ZrO₂, Rb₂O, SrO, and Nb₂O₅ assayed 0.021%, 0.014%, 0.014%, and 0.002% were identified, Table 1. Amounts of nickel, copper, and zinc oxides were detected, Table 2.

Size/chemical analyses of the original sample (as received) are shown in Table 3. It is clear that cassiterite showed concentration in SnO₂ reaching 0.39% – 0.25% in 0.59 – 0.074 mm fractions, Table 2. Fewer cassiterite concentrations assayed 0.17–0.11% SnO₂ were noted in coarser fractions 6.0- 0.83 mm,

and in the finer fraction – 0.074 mm, Table 3. On the other hand Table 3 illustrates the size/chemical analyses of the attrition product. It was noted that low content of fines below 0.074 mm reaching about 15% by wt., were produced after the attrition process, Table 4. The constitution of these fines besides cassiterite may include the soft minerals e.g. talc and clinochlore.

Results of sink/float tests using bromoform (sp.g. 2.89 g/cc) showed that 24.40% and 21.40% by weight of the attrition – 0.51 + 0.211 mm, and – 0.211 + 0.074 mm samples were separated as heavy products with increase in MgO from 10.97–13.38%, TiO₂ from 0.75–1.02%, Cr₂O₃ from 0.17–0.47%, and Fe₂O₃ from 8.73–14.38%, Table 5. On the other hand, the silica and alumina contents assayed 46.16% SiO₂, and 10.04% Al₂O₃, respectively, Table 5. The increase in content of these oxides may be attributed to the presence of tremolite, hornblende, and biotite granites in the sink fraction. Meanwhile, SnO₂ content increased from 0.199% in the original sample to 0.76% in the sink product, Table 5. This showed that almost all the cassiterite content in the original sample was concentrated as inclusions of different concentrations within the matrices of these heavy minerals.

Different samples cuts that were picked from the 6.0-2.28 mm original sample according to grain color were illustrated in Fig. 3. The petrography investigation of polished sections of these stones cuts showed that cassiterite inclusions were found almost throughout all the minerals matrices granitic formations in different concentrations and with various diameter sizes between 120 microns to 5 microns as shown in Fig. 4. as follows:

- Thin veins and grains of cassiterite (from 20 to 120 um) found in the fractured quartz Fig. 4. A.
- Patches of cassiterite (from 30 to 5 um in diameter) coating the surface of biotite, Fig. 4.B.
- Finely disseminating elongate crystals of cassiterite (from 10 to 5 um diameter) found in groundmass of Hornblende and Biotite, Fig. 4.C.
- Large crystal of cassiterite "Hexagonal" (150 um diameter), Fig. 4.D.
- Very large crystal of cassiterite (from 200–250 um diameter), Fig. 4.E.
- Large cassiterite particle (120 um diameter) and large vein of cassiterite found between biotite and hornblende (light brown in color), Fig. 4.F.
- Grains of cassiterite (from 40–120 um in diameters) found in the open space between quartz and microcline, Fig. 4.G.
- Grains of cassiterite (from 40–120 um in diameters) found in the open space between quartz and microcline (yellow brown in color), Fig. 4.H and I.
- Large grains of elongated cassiterite and topaz minerals were detected together in the 400 microns particle size, Fig. 4.J.

Table 2
XRF elemental analysis of the original
sample

Oxide	Wt%	Oxide	Wt%
Na ₂ O	0.615	NiO	0.083
MgO	10.967	CuO	0.011
Al ₂ O ₃	11.869	ZnO	0.008
SiO ₂	51.939	As ₂ O ₃	0.000
P ₂ O ₃	0.292	Rb ₂ O	0.014
SO ₃	0.031	SrO	0.014
K ₂ O	1.490	ZrO ₂	0.021
CaO	5.683	Nb ₂ O ₅	0.002
TiO ₂	0.754	SnO ₂	0.199
Cr ₂ O ₃	0.175	PbO	0.007
MnO	0.402	Cl	0.004
Fe ₂ O ₃	8.730		

Table 3
Size/chemical analysis of the original sample (as received)

Fraction, mm	Wt. %	Cum. Wt. %	SiO ₂ %	Al ₂ O ₃ %	Fe ₂ O ₃ %	SnO ₂ %
-6.0 + 1.168	14.60	14.60	47.66	5.25	7.66	0.17
-1.168 + 0.833	11.00	25.76	47.66	5.25	7.66	0.17
-0.83 + 0.598	10.10	36.66	45.52	5.65	7.39	0.38
-0.59 + 0.417	10.80	47.61	49.83	5.45	7.40	0.39
0.295	9.10	58.51	45.84	4.82	7.53	0.34
0.208	7.40	67.14	51.12	5.15	7.87	0.31
0.106	4.40	80.04	51.65	7.07	7.36	0.29
0.074	10.00	83.82	53.84	4.49	6.35	0.25
-0.074	22.60	100.00	46.18	5.37	5.91	0.11
Total	100.0		47.52		7.16	
Original	100.0		51.94	11.67	8.73	0.199

Table 4
Size/chemical analysis of the attrition sample

Fraction, mm	Wt. %	SiO ₂ %	Al ₂ O ₃ %	SnO ₂ %	Dist., %
-0.5 + 0.211	50.75	52.91	14.09	0.179	35.71
-0.211 + 0.074	11.56	55.52	6.91	0.239	10.85
Total	62.31			0.190	59.49
-0.074	37.59	51.62	8.77	0.21	40.51
Calculated	100.00			0.199	100
Original sample	100.00	51.94	11.87	0.199	100

Table 5
XRF analysis of sink fraction of the
attrition product – 0.51 + 0.074 mm

Oxide	Wt%	Oxide	Wt%
Na ₂ O	0.51	CuO	0.004
MgO	13.38	ZnO	0.012
Al ₂ O ₃	10.04	As ₂ O ₃	0.003
SiO ₂	46.19	Rb ₂ O	0.010
P ₂ O ₅	0.17	SrO	0.010
SO ₃	0.02	Y ₂ O ₃	0.004
K ₂ O	0.66	ZrO ₂	0.009
CaO	6.76	Nb ₂ O ₅	0/006
TiO ₂	1.02	SnO ₂	0.759
Cr ₂ O ₃	0.47	PbO	0/012
MnO	0.65	Cl	0.001
Fe ₂ O ₃	14.38	Br	0.012
NiO	0.104		

Cassiterite Enrichment Using Shaking Table

After applying some exploratory experiments on the shaking table separation of the 0.51 + 0.21 mm sample, it was noted that at table inclination 3°, and at constant wash water flow rate of 20 l/min., the gradual increase in stroke length from 2 cm, 2.5 cm, and to 3 cm was not accompanied by notable change in mineral recovery, yet an improvement in recovery reaching 82.5% at stroke length 2.5 cm was recorded, Fig. 5.

However, at table inclination of 4°, and the wash water flow rate of 20 l/min., no change in recovery (all recovery values were 78–80%. In addition, the results showed again some improvement to reach 85% at 2.5 cm stroke length, Fig. 5. At table inclination 5°, and water wash 20 l/min., again the same behavior as previously noted, no change in recovery (75% – 77%), and again the recovery showed improvement to 88% at 2.5 cm stroke length, Fig. 5. At constant table tilt 5° and stroke length 2.5 cm, the wash water flow rate seemed very effective on the mineral recovery. Gradual decreasing of water rate from 25 l/min. to 20

l/min., and 15 l/min., the recovery decreased from 89–84%, and then 76%, Table 6. At constant stroke length 2.5 cm and at low wash water flow rate at 15 l/min., the recovery gradually decreased from 81–76% by the gradual increase on the table tilt from 3° to 5°, Table 6.

At constant stroke length 2.5 cm and wash water flow rate 20 l/min., it was noted that the change in the table tilt showed no effect on the level of cassiterite recovery. Whereas by increasing the tilt from 4° to 5°, the recovery percentage remained un-changeable at the level of 85%, Table 6. At constant stroke length 2.5 cm, and wash water flow rate reaching 15 l/min, the recovery percentage decreased from 81–76% by decreasing the inclination from 4° to 5°. Meanwhile, at wash water flow rate reached 20 l/min., there was no change on the recovery% by changing the table tilt from 4° to 5°, Fig. 6. This information proved again that the wash water flow rate was a detrimental parameter on the table separation efficiency. As the wash water flow rate increased, the transport of gangue minerals to the tailings fraction increased which in turn improved the grade of the concentrate fraction. Moreover, higher grade of the concentrate fraction was obtained at higher level of deck tilt angle and feed flow-rate. With a lower level of deck tilt angle, an increase in the feed rate decreased the quality of the concentrate fraction.

However, the black to deep gray strip of heavy concentrate fraction was discharged over the far end of the deck, Fig. 7. In addition, great amount of middling mass composed of two distinguished colors (light gray and beige) were formed on the table surface. The gray mass was shown to contain most of the heavy minerals that constituted the original sample, i.e. hornblende, biotite, and tremolite, Fig. 7. Additionally, the middling beige in color mass was composed of light minerals which contained cassiterite inclusions i.e. quartz, and feldspars (albite, and microcline), Fig. 7. On the other hand, fragments of these light minerals that were nearly free from cassiterite inclusions were carried by the flowing water towards the tailings launder, Fig. 7. However, the variation in color between these cuts helped a lot in their identification.

It could be concluded that the coarse fraction – $0.5 + 0.21$ mm was stratified in a good manner at table inclination 5°, stroke length 2.5 cm, and table speed 280–300 rpm. In case of applying the finer fractions – $0.21 + 0.074$ mm, the suitable stroke length was reduced to 2 cm with increased table speed up to about 320–330 rpm. Usually the increase in the length of the stroke required a decrease in the number of strokes per minute and vice-versa to achieve efficient separation. High amplitude was necessary when treating relatively coarse particles in order to create complete dilation along with lower acceleration (Burt and Mills 1984). The Schematic Diagram showing the shaking tabling separation process was depicted in Fig. 7 (*N.B. in all figures the cassiterite grade% were multiplied by the factor 50 to facilitate the illustration*). The evaluation of the final end-products with respect to cassiterite recovery and grade after shaking table was illustrated in Fig. 8 and Table 6.

Table 6
Evaluation of shaking table products

Product	-0.51 + 0.211 mm			-0.211 + 0.074 mm			-0.51 + 0.074 mm	
	Wt.%	SnO ₂ %	SnO ₂ % Dist. _{opt.}	Wt.%	SnO ₂ %	SnO ₂ % Dist. _{opt.}	Wt.%	SnO ₂ % Dist., %
Heavy	2.66	1.45	20.30	2.10	1.98	18.08		
Middling 1	23.45	0.31	39.26	2.88	1.32	16.53		
Middling 2	36.34	0.20	38.25	69.22	0.19	65.39		
Total	62.45	0.29	97.81	74.20	0.28	91.61	40.27	0.29 96.94
Light tailing	37.55	0.01	1.98	27.15	—	—		
Calc. opt.	100.00	0.190	100.00	100.0	0.23	100		
Feed	50.75	0.190	47.75	11.56	0.23	11.45	62.31	0.19 60.49

Statistical Optimization Of Rer Magnetic Separation Process

The design summary and the factorial design results of the RER magnetic separation in terms of cassiterite recovery% as a response are shown in Tables 7 and 8. It can be seen that the cassiterite recovery% reached an optimum of 97% (runs 2, 5, 7, 8, 10, and 11) at splitter angle 72.50°, and belt speed 150.00 rpm. A regression Eq. (1) was obtained by multiple regression analysis of the experimental data as follows:

$$\text{Recovery\%} = -24.76 + 66.22*A + 0.68*B - 0.44*A^2 - 3.00E-004*B^2 - 8.00E-003*A*B \quad (1)$$

where, A is the splitter inclination angle, and B is the separator belt speed.

The optimization of Eq. (1) was performed using State-Ease program, by an iteration method. Statistical testing of the model has been carried out by F-test to produce ANOVA – the analysis of variance, Table 9. The values of R² and the standard deviation suggested that there was a good agreement between the experimental and predicted values obtained from the model, Fig. 9.

Results showed that with increasing the splitter inclination angle and the separator belt speed, cassiterite recovery value increased, and reached its maximum value of 97% at splitter angle 72.50° and at belt speed 150 rpm. However, it was evident that in these selected ranges of separator angle (70° – 75°) and belt speed (100–200 rpm), there was a dominant effect of the separator splitter inclination angle on cassiterite recovery % compared to the effect of belt speed, Fig. 10.

On the other hand, by increasing both splitter inclination and belt speed more than 72.50° and 150 rpm respectively, a slight decrease in the recovery value reaching 96% was remarked. Meanwhile, the decrease in the values of both variables to 70° and 100 rpm, respectively, a pronounced decrease in the cassiterite recovery value reaching 86% was remarked, Fig. 10. In addition, the interaction of both variables showed a reversible effect on the mineral recovery, Fig. 11. It was noticed that at low belt speed of 100 rpm, the increase in splitter angle was accompanied by gradual increase in cassiterite recovery from 86–92%, and then to 96%. However, at belt speed of 150 rpm, the change in splitter angle showed promising recovery reaching 90% at 70° , and then the recovery increased to reach 97% at splitter angles 75° . Additionally, the same trend was shown by increasing the belt speed to reach 200 rpm, Fig. 12. Response surface for cassiterite recovery% as function of RER magnetic separation variables, i.e. splitter angle, and belt speed was illustrated in Fig. 13.

An interesting study was carried out about the effect of feed size of both magnetic and nonmagnetic particles on the efficiency of the RER magnetic separator (Gehauf 2004). It was found that, when nonmagnetic particles travel over a roll and are allowed to drop unhindered, they are roughly classified by their particle size. Large particles will travel further from the centerline of the roll than smaller particles. Therefore, large particles are typically processed using lower surface speeds than small particles. When a magnetic roll is used, strongly attached magnetic particles will usually be pinned to the roll surface until they are released from the magnetic field. Weakly attached magnetic particles may only be deflected by the magnetic field, altering them from their normal path. When this occurs, there will be an overlap in the large weakly attached magnetic particles and the small nonmagnetic particles (Gehauf 2004). If the splitter is set to eliminate the large weakly attached magnetic particles, many of the small nonmagnetic particles will report to the magnetic product. On the other hand, if the splitter is set to recover the small nonmagnetic particles, the nonmagnetic product will contain many of the large weakly attached magnetic particles. This is an indication that the particle size range is too great. The problem can normally be overcome by screening the feed before magnetic separation and thus produce a better product. The practicality of screening is ultimately based on the difference in the magnetic responses of the particles to be separated and the value of the product(s). That was the way the present study started, i.e., feed fractionation into $-0.51 + 0.21$ mm and $-0.21 + 0.074$ mm (Ibrahim et al. 2017).

However, it could be concluded that the optimum separation conditions were reached by keeping the splitter angle fixed at 72.50° and by applying the belt speed at 150 rpm for coarse fraction and 200 rpm for finer fraction. At these conditions, the optimum operational concentration results of the $-0.50 + 0.21$ mm fraction was 2.10% wt., 12.96% SnO_2 , and 97.62% recovery from a feed containing 0.29% SnO_2 . On

the other hand, the optimum operational concentration results of the – 0.21 + 0.074 mm fraction was 3.72% wt., 6.21% SnO₂, and 90.00% recovery from a feed containing 0.28% SnO₂. From these results, it could be concluded that an overall final cassiterite concentrate contained 11.25% SnO₂, 2.29% wt, and 94.08% recovery from a feed contained 0.19% SnO₂, was produced, Fig. 14 (*N.B. in all figures the cassiterite grade% were multiplied by the factor 50 to facilitate the illustration*).

Important to mention that a non magnetic product containing topaz mineral was separated from the coarse fraction – 0.51 + 0.21 mm only at splitter angle of 65° and belt speed 150 rpm. This may be attributed to the presence of topaz that was impeded as inclusions within the hard quartzite matrix that resisted over-grinding and remained in the coarser size 0.50 + 0.21 mm. SEM/EDX and XRD analyses of topaz mineral, cassiterite/topaz, and cassiterite mineral are illustrated in Figs. 15–21. However, the schematic diagram for the suggested processing flow-sheet to recover cassiterite and topaz minerals from some mines scraps of the Eastern Desert of Egypt is illustrated in Fig. 22.

Table 7
Design summary

Study type		Response surface					
Experiments		13					
Initial design		Central composite					
Blocks		No blocks					
Design model		Quadratic					
Response	Name	Units	Obs	Minimum	Maximum	trans	Model
Y1	recovery	%	13	86.00	97.00	None	Quadratic
Factor	Name	Units	Type	Low actual	High actual	Low coded	High coded
A	Splitter angle	Degree	Numeric	70.00	75.00	-1.000	1.000
B	Belt speed	rpm	Numeric	100.00	200.00	-1.000	1.000

Table 8
Results of full factorial design

Std	Run	Block	Variable 1 A: Splitter angle, degree	Variable 2 B: belt speed, rpm	Response 1 Recovery %
3	1	Block 1	70.00	200.00	90.00
8	2	Block 1	72.50	220.71	97.00
2	3	Block 1	75.00	100.00	96.00
1	4	Block 1	70.00	100.00	86.00
9	5	Block 1	72.50	150.00	97.00
4	6	Block 1	75.00	200.00	96.00
12	7	Block 1	72.50	150.00	97.00
10	8	Block 1	72.50	150.00	97.00
6	9	Block 1	76.04	150.00	96.00
11	10	Block 1	72.50	150.00	97.00
13	11	Block 1	72.50	150.00	97.00
7	12	Block 1	72.50	79.29	86.00
5	13	Block 1	68.96	150.00	90.00

Table 9
ANOVA for response surface quadratic model in terms of recovery% as a response

Source	Sum of squares	DF	Mean square	F Value	Prob > F
Model	134.63	5	26.93	6.97	0.0121
<i>A</i>	<i>74.94</i>	1	74.94	19.39	0.0031
<i>B</i>	<i>2.00</i>	1	2.00	0.52	0.4593
<i>A</i> ²	<i>52.61</i>	1	52.61	13.61	0.0078
<i>B</i> ²	<i>3.91</i>	1	3.91	1.01	0.3479
<i>AB</i>	<i>4.00</i>	1	4.00	1.03	0.3429
Residual	27.06	7	3.87		
<i>Lack of Fit</i>	<i>27.06</i>	3	9.02		
<i>Pure Error</i>	0.000	4	<i>0.000</i>		
<i>Cor Total</i>	<i>161.69</i>	12			

Conclusions

A technological grab rock sample from the mines scraps of the Eastern Desert of Egypt was subjected to chemical and mineralogical investigation. The sample was below 6 mm in size. It was showed to contain 0.199% cassiterite mineral (SnO₂). Cassiterite was found as inclusions of different sizes from 250 microns to 5 microns. These inclusions were imbedded in granitic ground mass containing quartz-feldspar- hornblende-clinocllore-tremolite-biotite- and talc minerals in different concentrations.

Stirring ball milling was adapted to grind the sample into 100% -0.51 mm with minimum fines (as possible) to reach 15% by wt. of the feeding sample. However, the total fines below 0.074 mm of the original sample after attrition reached about 37% by wt. with about 38% cassiterite distribution. This fine product was kept aside for another beneficiation study.

The attrition – 0.51 + 0.074 mm product was shown to contain about 0.19% SnO₂ with cassiterite distribution reached 62% of the original sample. This product was divided into two fractions, -0.50 + 0.21 mm and – 0.211 + 0.074 mm. Each fraction was separately subjected to gravity separation using “Wilfley” shaking table. The coarse fraction – 0.5 + 0.21 mm was stratified in a good manner upon the table surface at table inclination 5°, stroke length 2.5 cm, and table speed in the range of 280–300 rpm. In case of applying the finer fractions – 0.21 + 0.074 mm, the stroke length and table inclination were reduced to 2 cm, and 4°, respectively, with increasing on table speed up to about 320–330 rpm. The overall table concentrate assayed 0.29% SnO₂ with a recovery reaching 96.94% from a feeding material containing 0.19% SnO₂.

The heavy and the two middling products of the two size fractions – 0.50 + 0.21 mm and – 0.21 + 0.074 mm were directed after dryness to dry high intensity magnetic separation using “Eriez” RER separator. At magnetic field strength approaching 21,000 gauss and feeding rate at 2 kg/ hr, mathematically designed experiments were used to test the statistical significance of the main operating parameters of the RER separator (i.e. splitter inclination angle and belt speed) on the separation efficiency.

Taking the main factors effects and their interactions into account, the optimum conditions were found to be 72.50° splitter inclination angle and 150 rpm, 200 rpm belt speed for the two size fractions – 0.50 + 0.21 mm and – 0.21 + 0.074 mm, respectively. Meanwhile, a high grade topaz mineral product was separated from the coarse fraction – 0.50 + 0.211 mm, as a non-magnetic fraction at splitter angle 65°. At these optimum conditions, a cassiterite concentrate of 11.25% SnO₂ with a recovery of 94.08% was obtained. Consequently, a high grade topaz mineral product was separated from the coarse fraction – 0.50 + 0.211 mm, as a non-magnetic fraction at splitter angle 65°. These results are highly imperative to achieve applicable processing flow-sheet of such kind of minerals’ secondary resources.

Declarations

The authors have no relevant financial or non-financial interests to disclose.

The authors have no conflicts of interest to declare that are relevant to the content of this article.

All authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this manuscript.

The authors have no financial or proprietary interests in any material discussed in this article.

References

1. Angadi SI, Sreenivas T, Jeon H-S, Baek S-H, Mishra BK (2015) A review of cassiterite beneficiation fundamentals and plant practices. *Minerals Engineering* 70: 178–200. <https://doi.org/10.1016/j.mineng.2014.09.009>
2. Ajaka EO, Akande JM, Saliu MA (2014) Design of comminution circuit for optimum performance of the gravity separation unit at intake iron ore processing plant, Nigeria. *Innovative Systems Design and Engineering* 5 (6): 28 – 30. ISSN 2222-1727
3. Burt B, Mills C (1984) *Gravity concentration technology*. New York: Elsevier
4. Baba AA, Yusuf AO, Ragi MA, et al (2020) Potential of a Nigerian cassiterite ore for industrial Steel coatings. *Rare Metal Technology* 201-208, Part of the Minerals, Metals & Materials Series book series (MMMS), TMS, Springer, Switzerland. <https://doi.org/10.1007/978-3-030-36758-9>
5. Bulatovic SM (2020) *Handbook of Flotation Reagents: Chemistry, Theory and Practice, Flotation of sulfide ores: Volume 1*. Elsevier Ltd.

6. Box GEP, Behnken DW (1960) Some new three level designs for the study of quantitative variables. *Technometrics* 2, 455.
7. Dimova T, Aprahamian B, Marinova M (2019) Research of the magnetic field inside a drum separator with permanent magnets. 16th Conference on Electrical Machines, Drives and Power Systems (ELMA). doi: <https://doi.org/10.1109/elma.2019.8771679>
8. Dalimunthe DY, Aldila H, Nuryadin A (2020) Optimization on the purification of cassiterite from low-grade cassiterite concentrate. *IOP Conf. Ser.: Earth Environ. Sci.* 599 012002: 1-6. doi:10.1088/1755-1315/599/1/012002
9. El Aref M, Abd El-Rahman Y, et al (2020) Mineral Resources in Egypt (I): Metallic Ores, The Geology of Egypt. © Springer Nature Switzerland AG 2020, Z. Hamimi et al. (eds.), *Regional Geology Reviews*, https://doi.org/10.1007/978-3-030-15265-9_14
10. Egbe EAP, Mudiare E, Abubakre OK, Ogunbajo MI (2013) Effectiveness of gravity separation methods for the beneficiation of Baban Tsauni (Nigeria) Lead Gold Ore. *International Journal of Scientific and Research Publications* 3 (5): 1-4. ISSN 2250-3153
11. EGSMA (1998) Metallogenic map of Arab Republic of Egypt, Metallic ores and non-metallic deposits, Scale 1:1,000,000. Egyptian geological survey and mining authority.
12. Falcon LM (1982) The gravity recovery of cassiterite. *Journal of the South Africa Institute of Mining and Metallurgy*: 112-117. ISSN 2250-3153
13. Gonzales LGV, Pino GAH, Torem ML (2013) Eletroflotation of cassiterite fines using a hydrophobic bacterium strain. *REM: R. Esc. Minas, Ouro Preto* 66 (4): 507-512.
14. Gehauf R (2004) A practical guide on selecting and optimizing rare earth magnetic separators. SME Annual Meeting, Denver, CO, USA.
15. Ibrahim SS, Farahat MM, Boulos TR (2017) Optimizing the performance of the RER magnetic separator for upgrading silica sands. *Particulate Science and Technology: An International Journal* 35 (1): 21-28, DOI: 10.1080/02726351.2015.1121179
16. Onyedika GO, Ogwuegbu MOC, Onuoha GN (2015) Energy potential of microwave heating: prior to and in leaching of low grade Kuru cassiterite ore in Jos, Nigeria. *FUTO Journal Series 1* (1): 78-83.
17. Hisayoshi K, Uyeda C, Terada K (2016) Magnetic separation of general solid particles realized by a permanent magnet. *Scientific Reports*: 6 (1). doi: <https://doi.org/10.1038/srep38431>
18. Jiao H, Shib C, Ruixia Tian R (2011) Research on Design and Magnet Assembly Process of Multivariate and Multi-Roll Permanent Magnetic Separator. *Advanced Materials Research* 201-203: 486-490. doi:10.4028/www.scientific.net/AMR.201-203.486
19. Kumar N, Joshi B and Asokan K (2019) The effects of thermal annealing on the structural and electrical properties of zinc tin oxide thin films for transparent conducting electrode applications. *Physica B: Condensed Matter* 558: 5-9, Elsevier
20. Lalasari LH, A Suharyanto A, Firdiyono F (2017) The effect of pretreatments on the dissolutions of impurities from Indonesian cassiterite mineral. *Journal of Physics: Conference Series* 817, 012064, Jakarta, Indonesia

21. Lungu, M. (2009) Separation of small nonferrous particles using a two successive steps eddy-current separator with permanent magnets. *International Journal of Mineral Processing* 93 (2): 172–178. doi: <https://doi.org/10.1016/j.minpro.2009.07.012>
22. Li Y, Yang F (2016) Research progress and development trend of permanent magnetic separators in China and Abroad. 3rd International Conference on Vehicle, Mechanical and Electrical Engineering (ICVMEE 2016), ISBN: 978-1-60595-370-0. <https://doi.org/10.12783/dtetr/icvme2016/4873>
23. Mouedhen I, Coudert L, Blais JF, Mercier G (2018) Study of factors involved in the gravimetric separation process to treat soil contaminated by municipal solid waste. *Journal of Environmental Management* 209: 23-36. <https://doi.org/10.1016/j.jenvman.2017.12.020>.
24. Nakahiro Y, Wakamatsu DT (1993) A study on the separation of fine cassiterite and quartz by liquid-liquid extraction. XVIII International Mineral Processing Congress, ISBN: 978-0-949106-81-0, page 9.
25. Nandi A, Mandal S, et al (2019) Application of hybrid rGO-ITO bilayer TCO on a-Si solar cell for performance enhancement. *IEEE J. Photovoltaics* 9: 12–7. DOI: 10.1109/JPHOTOV.2018.2873707
26. Slusarek B, Zakrzewski K (2012) Magnetic properties of permanent magnets for magnetic sensors working in wide range of temperature. *Przegląd elektrotechniczny (Electrical Review)* 88 (7), 123–126. ISSN 0033-2097
27. Saisinchai S, Boonpramote T, Meechumna P (2016) Recovery of fine cassiterite from tailing dump in Jarin tin mine, Thailand. *Engineering Journal* 20 (4). DOI:10.4186/ej.2016.20.4.41
28. Su Z, Zhang Y, Liu B, Lu M, Li G, Jiang T (2017) Extraction and separation of tin from tin-bearing secondary resources: A Review. *JOM* 69(11): 2364-2372.
29. Shvedchykova I, Melkonova I, Romanchenko J (2020) Research of magnetic distribution in the working area of disk separator, taking into account an influence of materials of permanent magnets. *EUREKA: Physics and Engineering*: 1.
30. Sreenivas T, Padmanabhan NPH (2002) Surface chemistry and flotation of cassiterite with alkyl hydroxamates. *Colloids and Surfaces A: Physicochemical and Engineering Aspects* 205 (1–2): 47-59. [https://doi.org/10.1016/S0927-7757\(01\)01146-3](https://doi.org/10.1016/S0927-7757(01)01146-3)
31. Tian M, Gao Z, et al (2018a) Selective flotation of cassiterite from calcite with salicylhydroxamic acid collector and carboxymethyl cellulose depressant. *Minerals* 8 (8): 316. <https://doi.org/10.3390/min8080316>
32. Tian M, Hu Y, Sun W, Liu R (2017b) Study on the mechanism and application of a novel collector complexes in cassiterite flotation. *Colloids and Surfaces A: Physicochemical and Engineering Aspect* 522: 635-641. <https://doi.org/10.1016/j.colsurfa.2017.02.051>
33. Tian M, Khoso SA, Wang L, Sun W, Zhang C, Hu Y (2019) Selective separation behavior and its molecular mechanism of cassiterite from quartz using cupferron as a novel flotation collector with a lower dosage of Pb²⁺ ions. *Appl. Surf. Sci.* 486: 228-238. <https://doi.org/10.1016/j.apsusc.2019.05.039>
34. Tian M, Liu R, et al (2018c) Activation mechanism of Fe (III) ions in cassiterite flotation with benzohydroxamic acid collector. *Miner. Eng.* 119: 31-37.

<https://doi.org/10.1016/j.mineng.2018.01.011>

35. Tian M, Zhang C, Han H, et al (2018e) Effects of the preassembly of benzohydroxamic acid with Fe (III) ions on its adsorption on cassiterite surface. *Miner. Eng.* 127: 32-41.
<https://doi.org/10.1016/j.mineng.2018.07.019>
36. Tripathy SK, Ramamurthy Y, et al (2010) Influence of shaking table process parameters on concentration of chromite plant tailings. In: *Proceedings of the XI International Seminar on Mineral Processing Technology*, Jamshedpur, India. © NML Jamshedpur, pp. 199–204
37. Pecina ET, Rodriguez M, Castillo P, Diaz V, Orrantia E (2009) Effect of *Leptospirillum ferrooxidans* on the flotation kinetics of sulphide ores. *Minerals Engineering* 22 (5): 462-468.
<https://doi.org/10.1016/j.mineng.2008.12.008>
38. Wills BA, Napier-Munn TJ (2006) *Mineral processing technology: an introduction to the practical aspects of ore treatment and mineral recovery*. Seventh Ed, by Heinemanns Ltd, Great Britain. Elsevier Science & Technology Books. ISBN: 0750644508
39. Yang J , Shuai Z, Zhou W, Ma S (2019) Grinding optimization of cassiterite-polymetallic sulfide ore. *Minerals* 9(2): 134. <https://doi.org/10.3390/min9020134>.
40. Yang JG, Wu YT, Zhang XL (2014) Study on separation of tin from a low-grade tin concentrate through leaching and low-temperature smelting processes. *Miner. Process. Extr. Metall.* 123: 228–33.
<https://doi.org/10.1179/1743285514Y.0000000070>
41. Zeng S, Zeng W, Ren L, An D, Li H (2015) Development of a high gradient permanent magnetic separator (HGPMS). *Minerals Engineering* 71: 21-26. <https://doi.org/10.1016/j.mineng.2014.10.009>.
42. Zhang L, Khoso SA, Tian M, Sun W (2021) Cassiterite recovery from a sulfide ore flotation tailing by combined gravity and flotation separations. *Physicochem. Probl. Miner. Process.* 57(1): 206-215.
DOI: 10.37190/ppmp/131006
43. Zhou Y, Tong X, Song S, Wang W (2014) Beneficiation of cassiterite fines from a tin tailing slime by froth flotation. *Sep. Sci. Technol.* 49: 458–463. <https://doi.org/10.1080/01496395.2013.818036>
44. Zong QX, Fu LZ, Bo L (2018) Variables and applications on dry magnetic separator. 3rd International Conference on advances in Energy and Environmental Research 53.
<https://doi.org/10.1051/e3sconf/20185302019>

Figures

Figure 1

Occurrences of cassiterite deposits in the Eastern Desert of Egypt (EGSMA 1998)

Figure 2

XRD analysis of the original sample

Figure 3

Major components of -6.0+2.23 mm original fraction

Figure 4

Petrography pictures of cassiterite inclusions within different granitic matrices

Figure 5

Effect of stroke length at different table inclinations on cassiterite recovery%

Figure 6

Effect of table inclination at different water flow rates on cassiterite recovery%

Figure 7

Schematic diagram showing the shaking tabling separation process

Figure 8

Schematic diagram showing the evaluation of the shaking tabling end- products with respect to cassiterite recovery and grade

Figure 9

Normal plot of residuals

Figure 10

Effect of belt speed at different splitter angles on cassiterite recovery%

Figure 11

Variables interaction effect on cassiterite recovery%

Figure 12

Effect of both variables on cassiterite recovery%

Figure 13

Response surfaces for cassiterite recovery% as function of RER magnetic separation variables

Figure 14

Schematic diagram showing the results of the dry RER magnetic separation processes of the coarse size fraction -0.50+0.21 mm

Figure 15

Element	Weight%
O K	38.04
F K	17.18
Mg K	0.33
Al K	25.21
Si K	16.96
K K	0.63
Fe K	0.25
Cu K	0.80
Zn K	0.59
Totals	100.00

SEM/EDX analyses of topaz mineral

Page 31/34

XRD analyses of topaz concentrate

Figure 18

SEM/EDX analyses of cassiterite/topaz minerals

Figure 19

XRD of cassiterite/topaz concentrate

Figure 20

SEM/EDX analyses of cassiterite mineral

Figure 21

XRD of cassiterite concentrate

Figure 22

Final processing flow-sheet