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DEVELOPMENT OF PROCESS FLOWSHEET TO RECOVER ILMENITE BY USING PROCESS MINERALOGY FROM LEAN GRADE BEACH PLACER SAND

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Abstract

This study focuses on the recovery of ilmenite and rutile from low-grade beach sand using a combination of gravity separation, "high-tension separation", and magnetic separation techniques. The analysis shows that the feed sample contains a total of 4.72% heavy minerals, with ilmenite constituting 1.56% and rutile 0.52%. A series of continuous experiments were carried out using various separation methods to effectively isolate ilmenite and rutile. Mineralogical modal analysis was performed to establish a flowsheet for the recovery of ilmenite and rutile. The results showed that ilmenite achieved a purity of 95.02%, whereas rutile achieved a grade of 82.9%.

Key words: *placer sand, titaniferous minerals, beneficiation, process mineralogy.*

1. Introduction

The Government of India has compiled an extensive inventory of more than 30 essential commodities designed to reduce reliance, bolster supply chain resilience, support re-employment efforts, and promote the country's goal of achieving zero net emissions. This newly published inventory aims to identify and monitor crucial materials across diverse sectors, including advanced electronics, telecommunications, defense, agriculture, energy, and healthcare. In line with its dedication to clean technology and the ambition of reaching net-zero greenhouse gas emissions by 2070, the government has released a policy statement. The inventory features a variety of minerals such as tungsten, titanium, potassium, phosphorus, niobium, nickel, molybdenum, lithium, indium, hafnium, graphite, germanium, gallium, copper, cobalt, bismuth, beryllium, antimony, and rare earth elements, among others. This article explores the importance and uses of titanium-rich minerals like ilmenite and rutile [1,2].

In India, the primary sources and trade of titanium ore are predominantly found in placer deposits along the coastlines. The distribution of these titanium placers varies from one coastal region to another, shaped by factors such as the underlying bedrock, the dynamics of river and wind transport, and the geographical features of the coast. The advent of nanotechnology has propelled titanium dioxide nanomaterials into the spotlight, owing to their cost-effectiveness and ease of production. This has resulted in an increasing demand for titanium dioxide nanoparticles, creating opportunities for businesses that offer these products. The global market for titanium dioxide nanomaterials can be segmented by their applications, which include personal care items, paints and coatings, energy solutions, paper and ink manufacturing, catalysts, and advanced filtration technologies. Furthermore, companies can capitalize on the advantages of titanium dioxide-based coatings for photovoltaic modules to improve their efficiency. As the photovoltaic and solar industries continue to expand rapidly, the demand for titanium dioxide in these applications is anticipated to rise significantly. This trend has prompted companies to make substantial investments in research and development, leading to a variety of new applications that are currently in the testing or development phases.

The classification of essential critical minerals differs

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across nations, influenced by their economic contexts and significance. The United States has recognized 50 critical minerals, while Japan has identified 31, the United Kingdom 18, the European Union 34, and Canada 31. India employs a three-phase approach to determine its list of 33 critical minerals [1]. Initially, they examined the primary strategies of other nations, including the USA and Japan, pinpointing 69 crucial aspects of the future plans of leading economies. The second phase involves extensive consultations with various government sectors, such as electrical energy, atomic energy, and renewable energy, to pinpoint minerals vital for specific projects. In the final phase, the evaluation of mineral significance is conducted using visual indicators created in collaboration with the International Energy Agency (IEA) [2]. This methodology aids in assessing the relevance of critical minerals tailored to India's distinct needs and priorities.

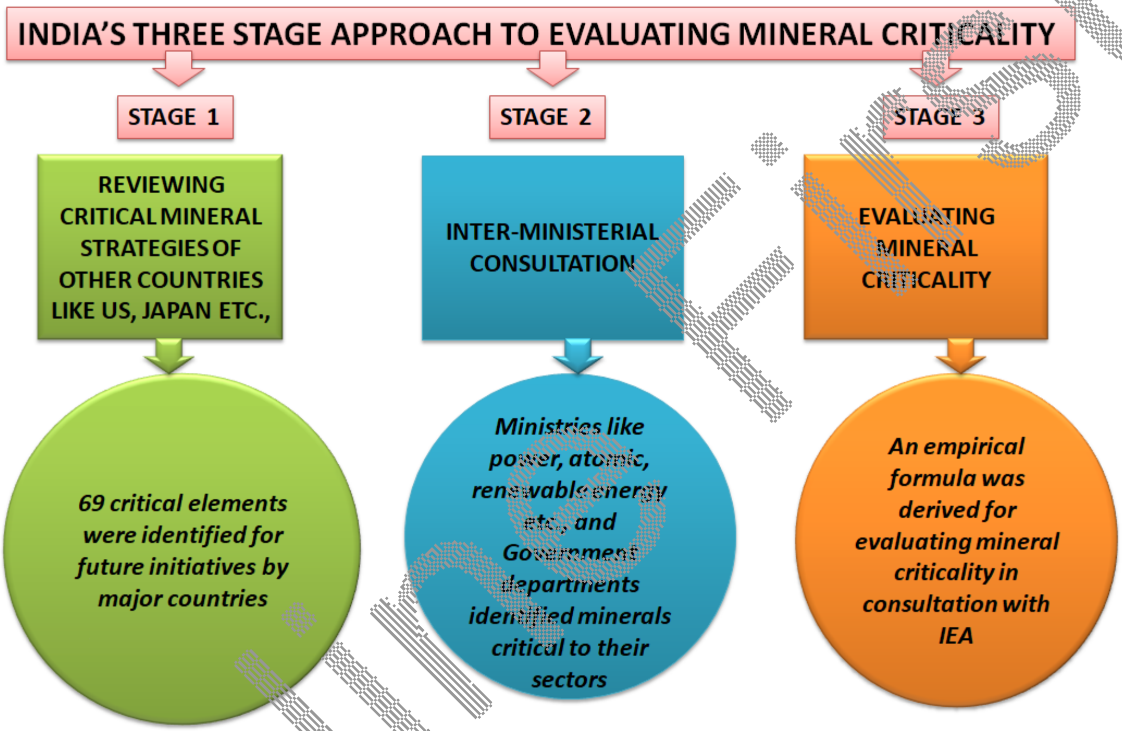


Figure 1 India's three stage approach for critical minerals [1]

In the present investigation, the critical miners which are available from beach sand, especially titanium minerals including ilmenite and rutile, are the priority to recover by simple physical beneficiation methods such as gravity separation followed by high tension separation and magnetic separation and the whole process flowsheet is designed with the help of process mineralogy.

2. Experimental

About 15 tons of beach sand sample was collected from Bramhagiri. Representative samples were prepared to analyze size and mineral composition. The CT spiral-concentrator test was utilized for roughing process, followed by cleaning with the HG8 spiral-concentrator. A typical recovery experiment is illustrated in Figure 2. CT Spiral and HG8 Spirals were operated at 20% solids concentration. Initially, all spiral products underwent sink-float studies using an organic liquid bromoform with a specific gravity of 2.89. The sink fraction contained titanium minerals, along with zircon, monazite, and sillimanite. The spiral material was continuously processed through HTS rougher unit. The Carpco High-Tension separator (Carrara HTR400 from MT Mineral Technologies) at 100 °C was used at one kg/hr to separate conducting and non-conducting minerals. The cleaner concentrate was then treated with the HTR Clean Separator. The final output from the HTRS underwent magnetic separation to isolate ilmenite as a magnetic material and rutile as a non-magnetic material. The magnetic separator used here was a Permoroll dry magnetic separator made of rare earth drums operated at 1.4T at one kg/hr. Leica petrological optical microscope (model no. Leica DM2500p) was used in plane-polarised and crossed-polarised lights to identify the minerals and counting the ilmenite and rutile minerals separately. The individual mineral weight, % was calculated by using the following formula (1):

$$\begin{matrix} Individual \\ grains \end{matrix} \cdot \begin{matrix} specific\ gravity \\ of\ the\ mineral \end{matrix} = \begin{matrix} mineral \\ weight\ \% \end{matrix} \quad (1)$$

Material calculations were conducted on all samples for mineralogical modal analysis to assess the recovery rates of ilmenite and rutile. Both fractions obtained are deemed suitable for industrial applications.

The wet chemical analysis methods were initially used for all products (mineral concentrates) and prepared standards. The inductively coupled plasma spectrophotometer PlasmaQuant 9100 ICP OES spectrometer from Analytik Jena was used to quantitatively analyze the elements present. The ASpect PQ software with an automatic baseline correction algorithm (ABC) and a tool for correcting spectral interferences (CSI) were also used. These standards are used for XRF. Elemental analyses of the samples were carried out by Philips (now PANalytical) PW2440 (MagiX PRO) sequential type wavelength dispersive X-ray spectrometer with Rh anode tube (operating at 4KW). Herzog HTP40 pelletisation press was used to prepare pellets. Herzog HAG 12 fusion machines were used for the fusion of the samples.

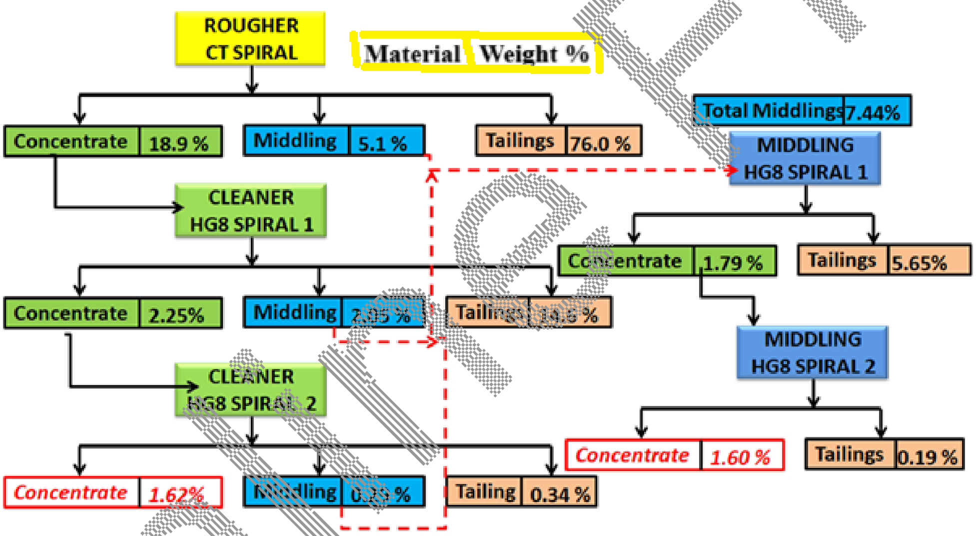


Figure 2 Mass distribution at various stages of spiral concentrators

3. Results and discussion

This study seeks to explore the process mineralogy associated with the recovery efficiency of placer titanium minerals, with the results analyzed in the context of leveraging process mineralogy to improve titanium mineral recovery. The Table contains data on mineralogical and chemical analysis of the feed sample. In Table 1 the heavy minerals, which comprise titaniferous minerals, rare earth minerals, abrasive, ceramic and refractory minerals, account for more than 4.7% of the sample.

These studies and the results are consistent with researchers who have undertaken considerable research efforts in the field of process mineralogy (using the mineralogical modal analysis method) over the past three decades [3-15], which include a variety of seminars. Nevertheless, the primary emphasis of this research has largely been on placer ilmenite or placer minerals [3-6], ilmenite ore [7-11] and review type or process mineralogy for sulphide ore or wastes for ilmenite recovery [12-15].

The total heavy and other minerals are accounting to 1.56% for ilmenite, 0.05% for rutile, 1.4% for garnet, 1.54% for sillimanite, 0.16% for zircon, and for quartz 95.28%. However, the overall percentage of significant minerals, particularly ilmenite and rutile, stands at 1.61%. In contrast, other heavy minerals, categorized as non-conducting minerals such as zircon, sillimanite, monazite and garnet, comprise 3.11%. Very heavy minerals account for 3.18%, with the light heavy mineral, sillimanite, contributing 1.54%. The total for magnetic heavy minerals such as monazite, garnet, ilmenite is 2.96%, while non-magnetic heavy minerals such as zircon, sillimanite, & rutile reach a total of 1.76%. Quartz is recognized as the dominant gangue mineral, constituting 95.28%. An analysis of particle sizes for both titaniferous and non-titaniferous minerals indicates that the d80 passing size for titaniferous minerals such as rutile and ilmenite is 190 µm, whereas non-titaniferous minerals, especially zircon, sillimanite, monazite, and garnet, exhibit a d80 passing size of 290 µm. The complete chemical analysis of the beach sand (Table 2) gives further information that the TiO_2 (ilmenite and rutile) contains 0.449%, ZrO_2 (Zircon) contains 0.039%, Al_2O_3 (Sillimanite) contains 2.196% and the presence of monazite by the analysis such as U_3O_8 8.21E-06% PbO 5.86E-06%, P_2O_5 0.003% and ThO_2 0.0004% clearly confirms that the monazite is present in traces only.

Table 1 The mineralogical and chemical properties of the feed sample

Table 1a Mineralogical analysis of the feed sample

Details	Percent	Minerals	Percent
Total heavy minerals in the bulk sample	4.72	Quartz	95.28
Total gangue minerals in the bulk sample	95.28	Ilmenite	1.56
Titaniferous heavy [conducting] minerals	1.61	Rutile	0.05
Total other heavy [non-conducting] minerals	3.11	Garnet	1.4
Total very heavy minerals	3.18	Sillimanite	1.54
Total light heavy minerals	1.54	Zircon	0.16
Total heaviest mineral [Monazite]	0.01	Monazite	0.01
Total magnetic heavy minerals	2.96	Total nonmagnetic heavy minerals	1.76
d80 passing size, for titaniferous minerals	190 µm	d80 passing size, non-titaniferous minerals	290 µm

Table 1b Chemical analysis of the feed sample

Oxides	[%]	Oxides	[%]	Oxides
TiO_2	0.449	FeO	0.873	Fe_2O_3
SiO_2	93.018	MnO	0.0770	Cr_2O_3
MgO	0.108	CaO	0.081	P_2O_5
U_3O_8	8.21E-06	PbO	5.86E-06	ZrO_2
Oxides	%	Oxides	%	Oxides
TiO_2	0.449	FeO	0.873	Fe_2O_3
SiO_2	93.018	MnO	0.0770	Cr_2O_3

This research focuses on the extraction of titanium-bearing placer minerals, particularly ilmenite and rutile, through a physical beneficiation method that employs a combination of gravity separation, high tension separation, and magnetic separation techniques. The efficacy of these processes is assessed using mineralogical modal analysis (process mineralogy) methods to develop a detailed flow sheet that includes a mineral/material balance.

The comprehensive mineralogical balance flow sheet for the pre-concentration of ilmenite and rutile, is achieved by strategically utilizing two types of spirals – CT-rougher spiral and HG8-cleaner spirals (Figures 3 and 4).

Figure 3 depicts the mineralogical characteristics involved in the processing of ilmenite for the recovery of total heavy minerals (THM) using CT-spiral and HG8-spirals. The findings suggest that to attain the target grade during spiral concentration, one rougher spiral, two-stage cleaner and two-stage middling cleaner spirals must be employed. Mineralogical data collected through grain counting is utilized to evaluate the flowsheet for ilmenite recovery. It is found that the CT rougher-spiral has an ilmenite concentration of 1.56%. The rougher concentrate generated has a yield of 18.9% and a mineral grade of 6.7%. This concentrate was further enhanced to

26.61% with a yield of 2.25% using the HG8 cleaner spiral. However, the final product, obtained from the two-stage rougher cleaner, middling cleaner, has an ilmenite concentration of 31.11%, with a recovery rate of 64.21%. The spiral product is subjected to the high-tension roller separator (Table 2). The resulting product achieved an ilmenite concentration of 95.02%, with a yield of 1.04% and a recovery rate of 63.35%. This product is recognized as suitable for industrial use.

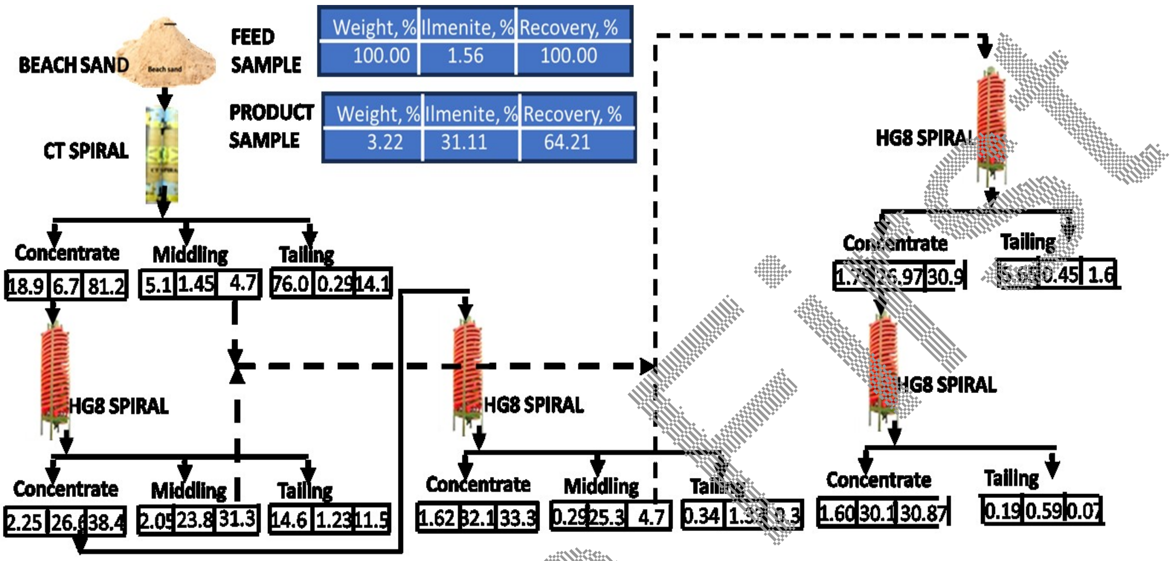


Figure 3 Mineralogical analysis of ilmenite in the recovery of total heavy minerals using CT rougher and HG8 cleaner spiral concentrators

Table 2 Mineralogical data for ilmenite (1.56% in feed and for HTRS 31.11%) and rutile (0.05% in feed and for HTRS 1.04%) obtained from the High -Tension Roller and magnetic Separators

HTRS	Products	Weight [%]	Ilmenite		Rutile	
			Grade [%]	Recovery [%]	Grade [%]	Recovery [%]
HTRS Rougher	Conducting	1.24	79.8	63.43	2.67	66
	Non Conducting	1.98	0.62	0.78	0.03	01
	Total	3.22	31.11	64.21	1.04	67
HTRS Cleaner	Conducting	1.04	95.02	63.35	3.18	66
	Non Conducting	0.20	0.67	0.08	0.03	0.1
	Total	1.24	79.8	63.43	2.67	66
Magnetic separation	Magnetics	0.02	98.70	1.3	0.002	-
	Non magnetics	1.02	94.95	62.1	3.25*	66
	Total	1.04	95.02	63.4	3.18	66

*Further processed [Table 4]

The process of extracting rutile from total heavy minerals using CT rougher and HG8 cleaner spiral concentrators is depicted in Figure 4 and Table 4. This procedure follows a similar flowsheet that used for ilmenite recovery. The results indicate that 82.9% rutile can be achieved with a yield of 0.04% and a recovery rate of 66%, employing a combination of spirals, high-tension rollers, and magnetic separators (see Table 3). It is seen as expected that the rutile, a conducting mineral, is almost negligible in non-conducting fraction as per process mineralogy studies. Similarly in magnetic fraction, the presence of the nonmagnetic rutile is negligible.

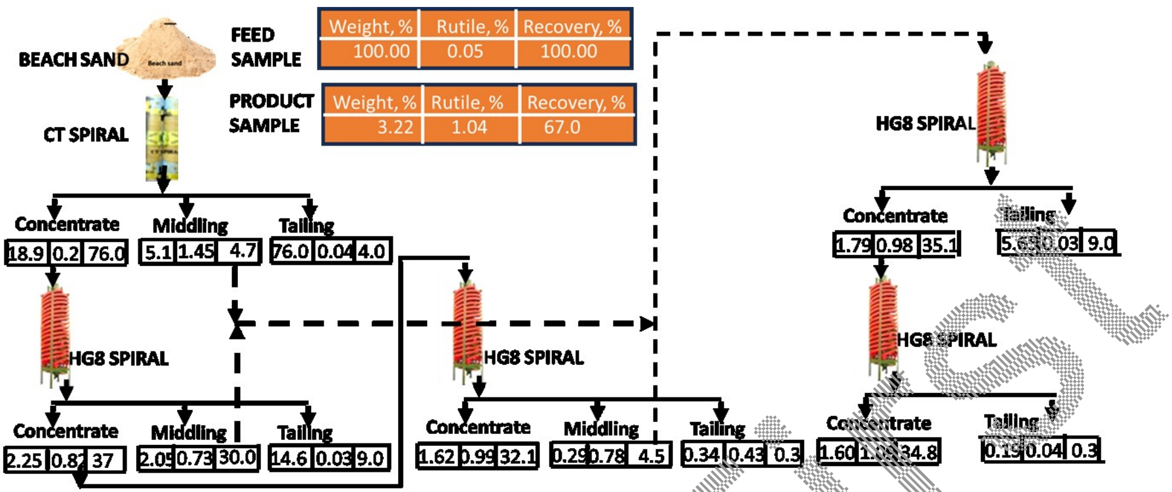


Figure 4 Rutile process mineralogy in recovery of total heavy minerals using CT rougher and HG8 cleaner spiral concentrators

Table 3 Results of the cleaning process for the non-magnetic product aimed at rutile recovery from the ilmenite circuit (see Table 2).

Magnetic Products	Weight [%]	Mineral grade [%]	Recovery [%]
Magnetic	0.98	0.003	-
Non Magnetic	0.04	82.90	66
Total	1.02	3.25	66

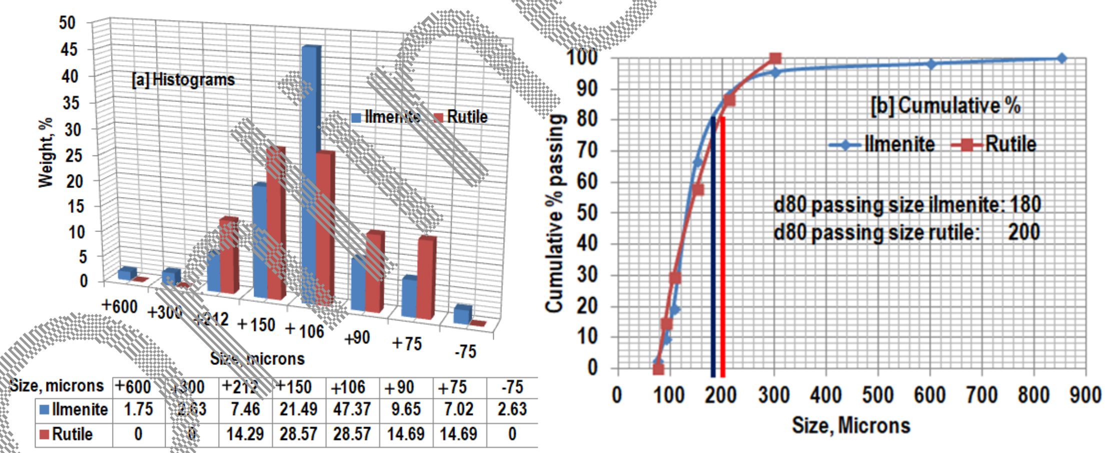
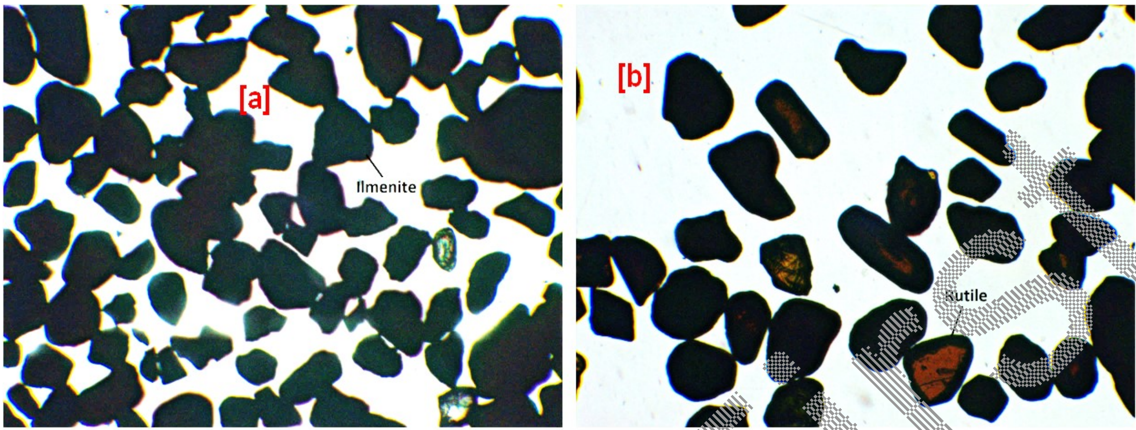


Figure 5 Size analysis of ilmenite and rutile in the spiral feed sample

The analysis of the particle sizes of titanium minerals, particularly ilmenite and rutile, which are utilized as inputs for spiral concentrators as depicted in Figure 5, indicates that ilmenite is mainly found in the -212+150 µm size range, accounting for 43.37% by weight. Conversely, rutile is present in equal concentrations of 42.94% in both the -212+150 µm (14.69%) and -150+106 µm size ranges (28.25%). In the size fraction below 106 µm, rutile demonstrates a slightly elevated concentration. Overall, the average content of rutile is considerably lower than that of ilmenite in the feed sample. The size analysis suggests that rutile is marginally coarser than ilmenite in the smaller size ranges, although both minerals display a comparable pattern in their size distribution.



a) ilmenite mineral grains b) rutile mineral grains

Figure 6 Morphological images of ilmenite and rutile [titanium critical minerals]

Figure 5 illustrates the size analysis of ilmenite and rutile within the spiral feed sample. The morphological characteristics of ilmenite and rutile, as depicted in Figure 6, indicate that ilmenite presents an angular to conchoidal morphology, whereas rutile grains are mainly sub-rounded to rounded, with some exhibiting elongated shapes and smooth edge contours. From the analysis of size and morphology, it can be deduced that although both ilmenite and rutile are conductive minerals, ilmenite, noted for its magnetic properties and relatively larger size distribution, has been favored for separation in the processes of spiral concentration, electrostatic separation, and magnetic separation. As a result, the outcomes from these techniques show that the ilmenite grade achieved is 95.02%, with a yield of 1.04% and a recovery rate of 63.35% from a feed sample containing 1.56% ilmenite. In comparison, the rutile grade obtained is 82.9%, with a yield of 0.04% and a recovery of 66% from a feed sample containing 0.05% rutile.

4. Conclusions

The following conclusions are drawn from the mineralogical modal analysis performed on the feed sample intended for the spiral concentrator and extraction of critical placer titanium minerals, namely ilmenite and rutile, through a sequence of gravity, high tension, and magnetic separators:

- The feed sample contains 1.56% ilmenite, 0.05% rutile and a total of 4.72% heavy minerals.
- The total of titaniferous minerals, which include both ilmenite and rutile, account for a total of 1.61%, while the remaining heavy minerals, which are classified as non-conductive, account for 3.11%.
- Very heavy minerals (VHM) account for 3.18%, while the light heavy mineral (LHM) sillimanite accounts for 1.54%.
- Total magnetic heavy minerals (TMHM) are noted at 2.96%, while total non-magnetic heavy minerals are at 1.76%. The d80 passing size for the titaniferous minerals is 190 μm.
- The spiral, electrostatic and magnetic separation results show that the ilmenite grade obtained is 95.02%. The recovery is limited to 63.35 with yield of 1.04%.
- The rutile grade, on the other hand, is 82.9%, and the recovery is 66%.

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Conflict of interest

There is no conflict of interests or publishing the sensitive data.

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5. References

[1] Joshi, P. (2023), Critical minerals for India; Report of the Committee on Identification of Critical Minerals, Report by Ministry of Mines, Government of India, Union Minister Pralhad Joshi unveils List of "Critical Minerals for India", (accessed 30. 1. 2025.)

[2] International Energy Agency, (2023), Critical Minerals Market Review, Critical Minerals Market Review 2023 – Analysis - IEA, (accessed 30. 1. 2025.)

[3] Singh, D., Mishra, B. R., Basu, S. & Rao, R. B., (2023) Process Mineralogy for the Development of a Flowsheet to Recover Monazite from Offshore Placer Deposit, Journal of The Institution of Engineers (India): Series D, 105, 477-487, https://doi.org/10.1007/s40033-023-00487-6

[4] Rao, R. B., Mishra, B., & Singh, D. (2024) Recovery of Rare Earth Element-Bearing Placer Minerals. Journal of Mining Science, 60 (1), 163-170.

[5] Philander, C., & Rozendaal, A. (2014) A process mineralogy approach to geometallurgical model refinement for the Namakwa Sands heavy minerals operations, west coast of South Africa. Minerals Engineering, 65, 9-16.

[6] Xianzhi, L., Peiyao, L., Yingjie, C., Zhan, D., Pan, Y., & Shaojun, B. (2022) Study on process mineralogy of ilmenite in Yunnan Province. Multipurpose Utilization of Mineral Resources, (2), 206-210.

[7] Mangeng, L. X. Z., (2009) Process Mineralogy Research on the Titanium Concentrate from a Mining Field Panxi Region Multipurpose Utilization of Mineral Resources, (1), 24-24.

[8] Zhu, F., Ma, Z., Gao, C., Qiu, K., & Peng, W. (2023) Process mineralogy of vanadium titanomagnetite ore in Panzhihua, China. Separations, 10 (3), 147, 1-20.

[9] Wen-Lin, N., Qian, Z., Xiao-Yong, Y., Qi-Cheng, F., Shu-Ming, W., Yao-Wen, Z., Jun-Bo, L. & Xiu-Zhu, Y. (2021) A study of the process mineralogy of vanadium-titanium magnetite electric furnace slag. Acta Petrologica Et Mineralogica, 40 (3), 542-550.

[10] Ying, J., Bo, L., Dongyun, L., & Lili, Z. (2020). Process Mineralogy Study on a Weathered Clay Type Titanium Ore Deposit. Multipurpose Utilization of Mineral Resources, 41 (6), 31-36.

[11] Bonifazi, G., & Gorga, R. (1994) Digital process mineralogy applied to titanium minerals characterization. In: 1994 AIME-SME Annual Meeting and Exhibit, Albuquerque, New Mexico, USA, Process Mineralogy of Titanium: Exploration and Processing, 1-15.

[12] Zhang, J. H., Li, C. X., & Zeng, L. X. (2013) Study on process mineralogy and titanium separation of Ti-bearing EAF slag. Advanced Materials Research, 734, 1097-1103.

[13] Li, Z., Xu, Y., Wang, J., Song, X., & Yu, J. (2015) Process mineralogy analysis of waste residue from ilmenite by acid hydrolysis. CIESC Journal, 66 (5), 1947-1954.

[14] Gan, F., Zhang, Y., Ruan, D., & Peng, X. (2020) The Application of Process Mineralogy Research in the Reasonable Utilization of Mineral Resources. In: 2020 5th International Conference on Smart Grid and Electrical Automation (ICSGEA), Zhangjiajie, China, 631-634, https://doi.org/10.1109/ICSGEA51094.2020.00143

[15] Bradshaw, D. (2014) The role of process mineralogy in improving the process performance of complex sulphide ores. In: XXVII International Mineral Processing Congress, Santiago, Chile, Proceedings of the XXVII International Mineral Processing Congress, 932: 1-25.

DEFINISANJE TEHNOLOŠKE ŠEME ZA ISKORIŠĆENJE ILMENITA IZ NISKOKVALITETNOG PESKA SA PLAŽE

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Izvod

Ova studija se fokusira na iskorišćenje ilmenita i rutila iz niskokvalitetnog peska sa plaže primenom kombinacije gravitacijske separacije, visokonaponske elektrostatičke separacije i magnetne separacije. Analiza je pokazala da ispitivani uzorak peska sadrži ukupno 4,72% teških minerala, pri čemu ilmenit čini 1,56%, a rutil 0,52%. Sproveden je niz kontinuiranih eksperimenata gde su korišćene različite metode separacije kako bi se efikasno valorizovali ilmenit i rutil. Mineraloška analiza izvršena je u cilju definisanja tehnološke šeme za valorizaciju ovih minerala. Rezultati su pokazali da koncentrat sadrži 95,02% ilmenita i 82,9% rutila.

Ključne reči: *pesak sa plaže, minerali titanijuma, obogaćivanje, procesna mineralogija.*