

Preparation of Nano Materials from Strategic Placer Heavy Minerals Recovered from Red Sediments of Badlands Topography of South East Coast of India

**Bignaraj Mishra¹, Satya Sai Srikant^{2*}, Sunita Routray³,
Tumula Laxmi⁴, Raghupatruni Bhima Rao⁵**

¹ Deputy General Manager , IREL, Chatrapur, India

^{2*} SRM Institute of Science and Technology, Modinagar, India

³ C V Raman College of Engineering, Bhubaneswar, India

⁴ The Techno School, Bhubaneswar, India

⁵ Former Chief Scientist, CSIR-IMMT, Bhubaneswar, India

Corresponding Author: Satya Sai Srikant

Corresponding E-mail Id: satya.srikant@gmail.com

Preparation of Nano Materials from Strategic Placer Heavy Minerals Recovered from Red Sediments of Badlands Topography of South East Coast of India

Abstract.

This paper deals with mine to metals and materials especially preparation of nano materials from strategic minerals of placer deposits which are derived from the badlands of topography exists along the coastal line of Eastern part of India. In the present investigation, red sediment samples are collected from the badlands and subjected to physical separation processes to recover high grade individual placer heavy minerals for value addition, which includes preparation of titanium oxide, titania slag, titanium oxide nano materials from comminuated ilmenite mineral as well as preparation of zircon flour, zirconia nano materials from natural zircon mineral.

Keywords: ilmenite, zircon, nano materials, badlands topography, beneficiation, leaching.

1. Introduction

Badlands topography exists all along the coastal line of Eastern part of India. These badlands are the recent deposits as per geological time scale. The topography of badlands depends on the geological locations. However, all the badlands release red sediments along with fluvial placer minerals during rainy season. The placer minerals which consist of ilmenite, sillimanite, zircon, monazite, rutile etc., occur along the fluvial deposits with varying mineral composition and size range [1-2]. The recovery processes for individual minerals from these badlands are varying based on the limitations of ferrous coating, particle size range and presence of garnet. Atomic Minerals Division, Department of Atomic Energy has done detailed exploration work and characterisation on mineral deposits of badlands. The CSIR, IMMT has developed the process flow sheet to recover individual heavy minerals and value addition of minerals especially for ilmenite, zircon and sillimanite, which includes preparation of titanium oxide, titania slag, titanium oxide nano materials from comminuated ilmenite mineral as well as zircon flour, zirconia nano-materials from natural size zircon mineral.

Since the advent of nanotechnology, titanium dioxide nano-materials have been at the centre of research owing to their low cost and simple production process. Thus, the titanium dioxide nano-particles are witnessing higher demand; companies offering such solutions can capitalize on the emerging demand trends. The global titanium dioxide nano materials market can be divided on the basis of application like personal care products, paints and coatings, energy, paper and ink manufacturing, catalysts, and others including the creation of advanced water filters [3-4]. Another opportunity that companies can capitalize on the increasing use of titanium-dioxide-based coatings in photovoltaic modules so as to improve the operational efficiency of such modules. With the photovoltaic and solar energy market [5] demonstrating exceptional growth rates, the demand for titanium dioxide in this application will be high. Hence, manufacturers are investing heavily in R&D and have several new applications in their pilot or development phases.

Zirconium oxide nano-particles (ZrO_2) are available in the form of nano-dots, nano-fluids and nano-crystals having a wide surface area. They are often doped with either yttrium oxide or calcia or magnesia to stabilise in high temperature crystalline phase such as tetragonal or cubic phase [6]. The zirconium oxide nano-particles are applied in (a) ceramics for making ceramic pigments, porcelain glaze, etc., (b) making artificial jewelers; (c) making abrasive, insulating and fire-retarding materials; (d) the powder containing pyro-optical properties and hence used for optical storage, light shutters and stereo television glasses [7-9].

The present study deals with the typical badlands of South East coast of India. The placer minerals released from these badlands consists of ilmenite, sillimanite, zircon, traces of monazite and free from garnet. So far, no attempt has been made in India or elsewhere except CSIR-IMMT, Bhubaneswar on these typical badlands, for recovery of individual placer heavy minerals and its value addition especially for nano-materials [10]. The results obtained from mines to nano materials are suitable for industrialisation.

2. Materials and methods

2.1 Raw Materials

Red sediments samples were collected from typical Badlands Topography along the South East Coast of India [A] Bhimunipatnam, Visakhapatnam District, [B] Vastavalasa, Srikakulam District (found 6m below of the beach sand deposit) and [C] Basanputti, Ganjam District [2,10]. Initially, the red sediment samples of all the three location were deslimed and subjected to size analysis and sink float studies. The heavy minerals obtained from bromoform sinks was also subjected to magnetic separation using permanent drum magnetic separator at magnetic intensity of 1.2 T. Mineralogical modal analyses of all these three samples were carried out using binocular microscope.

2.2 Recovery of Heavy minerals

Recovery of total heavy minerals has been carried out for 100 tons of samples using desliming units and gravity spirals. Recovery of ilmenite minerals is carried out from the recovery of total heavy minerals. The total heavy minerals are subjected to wet high intensity magnetic separator. The magnetic minerals were further subjected to high tension roller separator for recovery of ilmenite minerals. Zircon minerals were recovered from total non- magnetic minerals via stage high tension separator.

2.3 Value addition

Initially, the recovered ilmenite sample was subjected to process of roasting followed by leaching for preparation of synthetic rutile. Ilmenite sample was also ground to different sizes and subjected to above leaching processes. Further pre oxidized ilmenite mineral concentrate was subjected to microwave heating furnace and developed titania slag and iron metal. At the end, an attempt had also been made to prepare nano-materials from ilmenite concentrate using sulphuric acid [11]. The experimental plan for preparation of nano Titania (TiO_2) material is shown in Fig .1 with flow sheet.

Zircon (as mined) obtained after mineral separation was fused with caustic flakes at 600-650 °C and leached with water to separate silica as soluble sodium silicate [12]for preparation of nano materials. Required doping was done by using a suitable stabilising oxide at mixing stage to get stabilised zirconium oxide either in cubic or tetragonal form [13]. The experimental plan for preparation of nano zirconium oxide material is shown in flow sheet (Fig. 2).

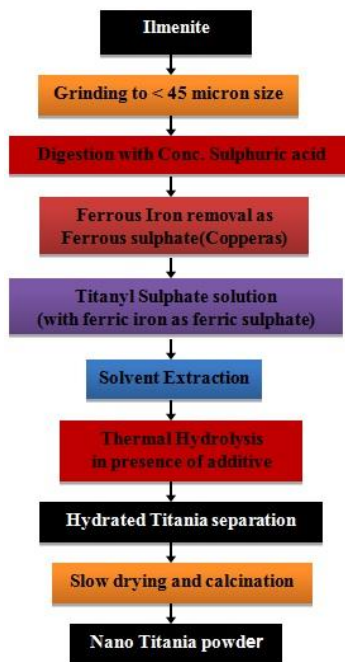


Fig 1. Preparation of Nano Titania material

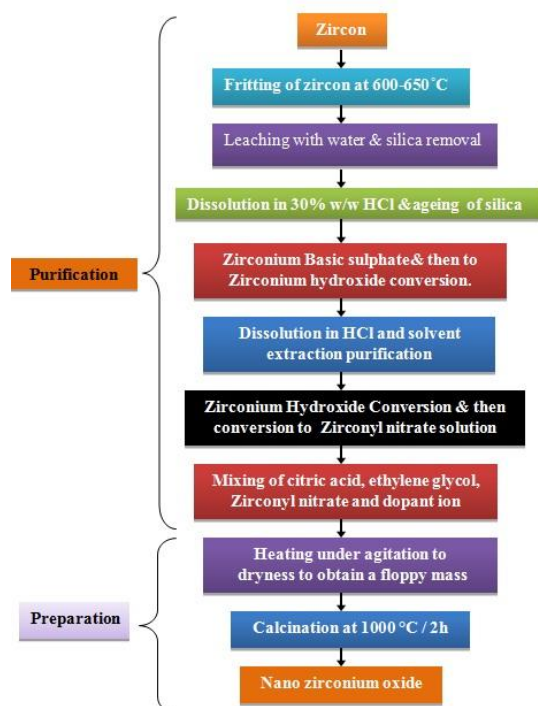


Fig 2. Preparation of Nano zirconium oxide

3. Results and Discussions

3.1 Modal and Size Analysis of Red sediments samples

Typical Badlands Topography along the South East Coast of India are shown in Fig.3 as Fig.3(A) Bhimunipatnam, Visakhapatnam District, Fig.3(B) Vastavalasa, Srikakulam District (found 6m below of the

beach sand deposit) and Fig.3(C) Basanputti, Chatrapur District. It can be seen from Fig. 3 that geomorphological formations of badlands located in different places are depend on the climatic conditions and geological settings as well as geological time scale.



Fig 3. Typical Badlands Topography along the South East Coast India (A) Bhimunipatnam, Visakhapatnam District; (B) Vastavalasa, Srikakulam District (found 6m below of the beach sand deposit) and (C)Basanputti, Ganjam District

Mineralogical modal analysis of red sediment samples collected from each location is shown in Fig. 4. The data indicate that the strategic minerals present in each deposit (A) Bhimunipatnam, Visakhapatnam District of Andhra Pradesh state contain ilmenite 71.2% and zircon contain 4.5% by weight, (B) Vastavalasa, Srikakulam District (found 6m below of the beach sand deposit) of Andhra Pradesh state contain ilmenite 70.3% and zircon contain 3.7% by weight, and (C) Basanputti, Ganjam District of Odisha state contain ilmenite 86.5% and zircon contain 1% by weight.

The deslimed feed size analysis as well as total heavy minerals, total very heavy minerals, total light heavy minerals, total magnetic heavy minerals and total non-magnetic heavy minerals size analysis of red sediment samples [14-15] of each sample location are shown in Fig. 5, Fig. 6 and Fig. 7. The data indicate that there is a distinct difference in size analysis of all three deposits, but with reference to the mineral processing equipments such as cyclones for desliming, gravity spirals, gravity tables, magnetic and electrostatic separator used for separation of individual minerals, the difference in size range in all three deposits are same.

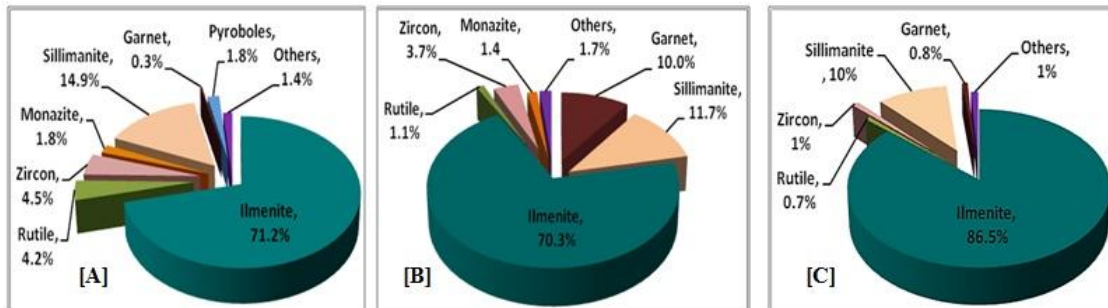


Fig 4. Modal analysis of heavy minerals present in red sediments of badlands topography. (A) Bhimunipatnam, (B) Vastavalasa and (C) Basanputti [14-15]

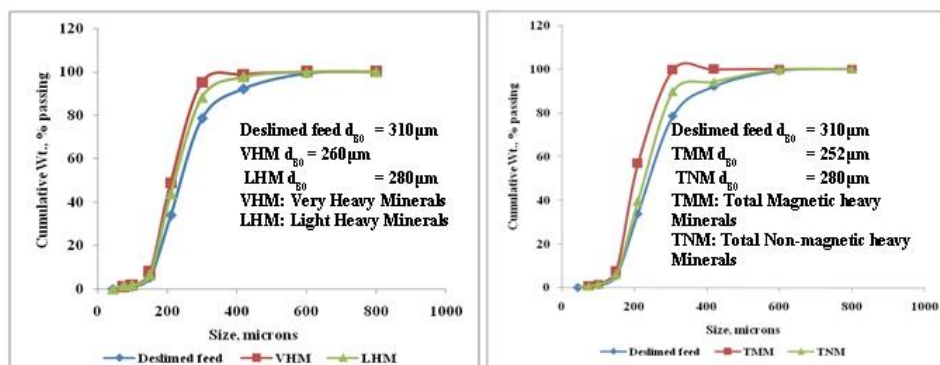


Fig 5. Size analysis of deslimed feed, VHM, LHM, TMM and TNM of red sediments of Bhimunipatnam

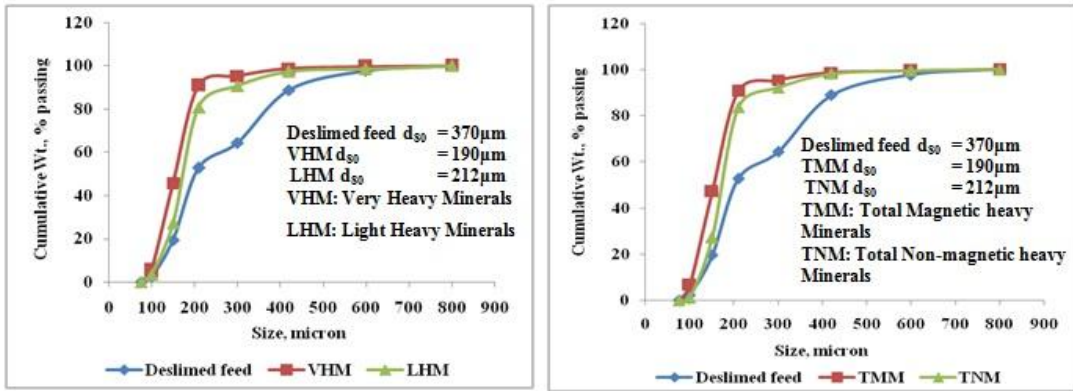


Fig 6. Size analysis of deslimed feed, VHM, LHM, TMM and TNM of red sediments of Vastavalasa

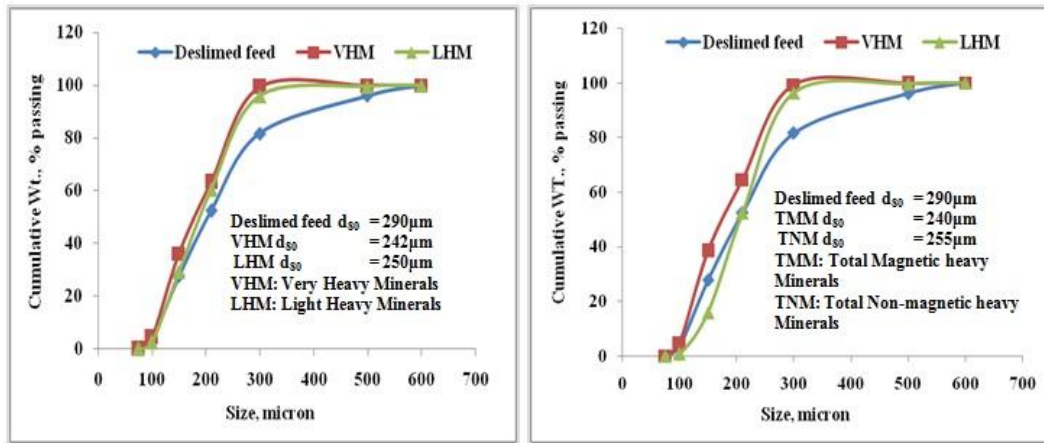


Fig 7. Size analysis of deslimed feed, VHM, LHM, TMM and TNM of red sediments of Basanputti

It is very clear from the data presented in Fig 4, Fig 5, Fig 6 and Fig 7 that the Odisha coastal belt red sediments of badlands topography contain more ilmenite (86.5% by weight). Hence from point of view of ilmenite content as well as transport of 100 tons of sample for processing, the Odisha coastal belt red sediment fluvial deposit sample (contain 33.2% THM) is chosen for mineral separation plant followed by recovery of ilmenite and zircon for value addition studies. The bulk sample is subjected to de-sliming unit to recover total sand. This sand is subjected to 7 stage spirals to recover total heavy minerals as shown in Fig. 8 [15].

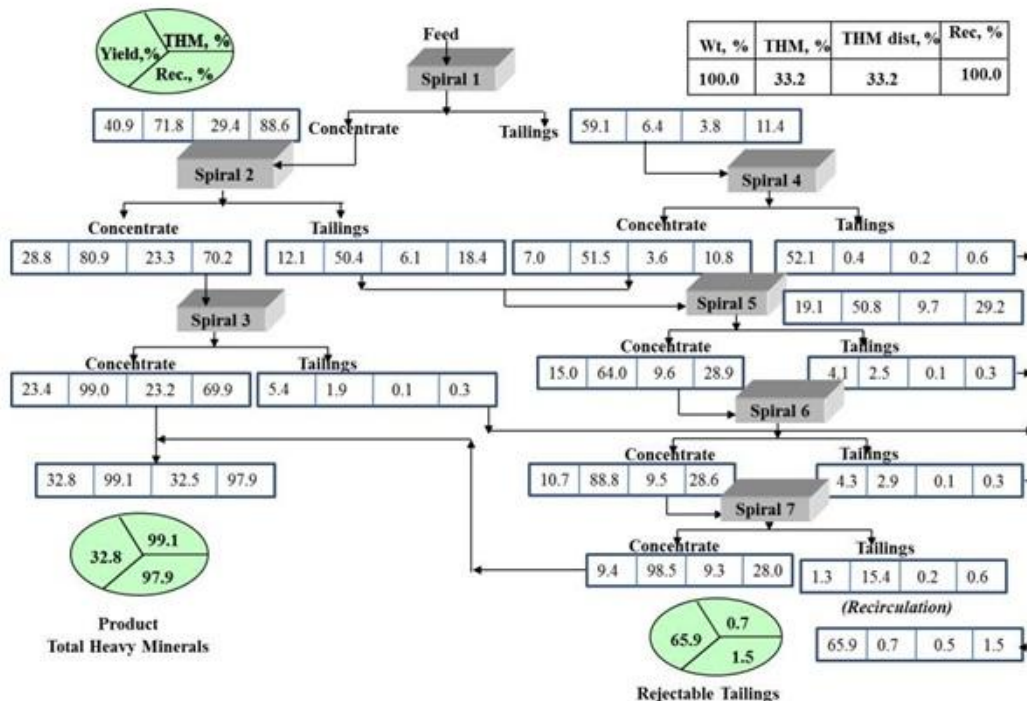


Fig 8. Flow sheet with material balance on recovery of total heavy minerals from red sediments of Basanputti village, Ganjam District Odisha (India) by using seven stage spirals

The data indicate that total heavy mineral concentrate contain 99.1% THM, with 97.9% recovery from feed sample contain 33.2% THM.

3.2 Recovery of strategic minerals

The recovery of total heavy minerals as shown in Fig 8, are subjected to initially wet high intensity magnetic separator to recover total magnetic minerals including ilmenite, monazite and other traces of garnet and pyribole minerals. These total magnetic minerals are subjected to high tension separator for recovery of conducting minerals ilmenite as shown in Fig 9. The data indicate that 99.7% ilmenite is recovered with 89% recovery from a deslimed feed contain 48.5% ilmenite. The non-magnetic minerals, obtained from ilmenite (as in Fig 9) are subjected to gravity tables to recovery heavy minerals.

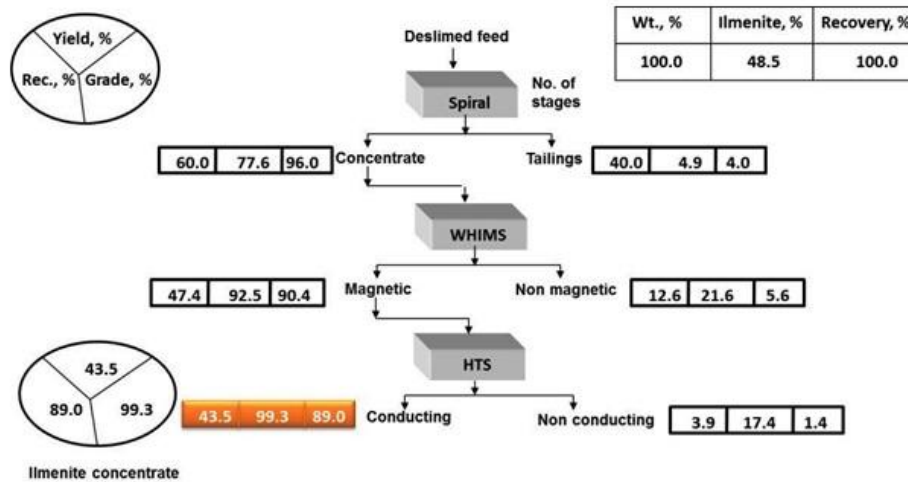


Fig 9. Flow sheet with material balance on recovery of ilmenite from deslimed red sediments sample of Odisha coast

The deslimed recovered heavy minerals is subjected to two stage flotation to remove sillimanite mineral and get other minerals concentrate which contain mostly zircon and rutile (in tailings). The flotation tailings subjected to two stage high tension roller separator to recover non-conducting zircon mineral concentrate as shown in Fig 10.

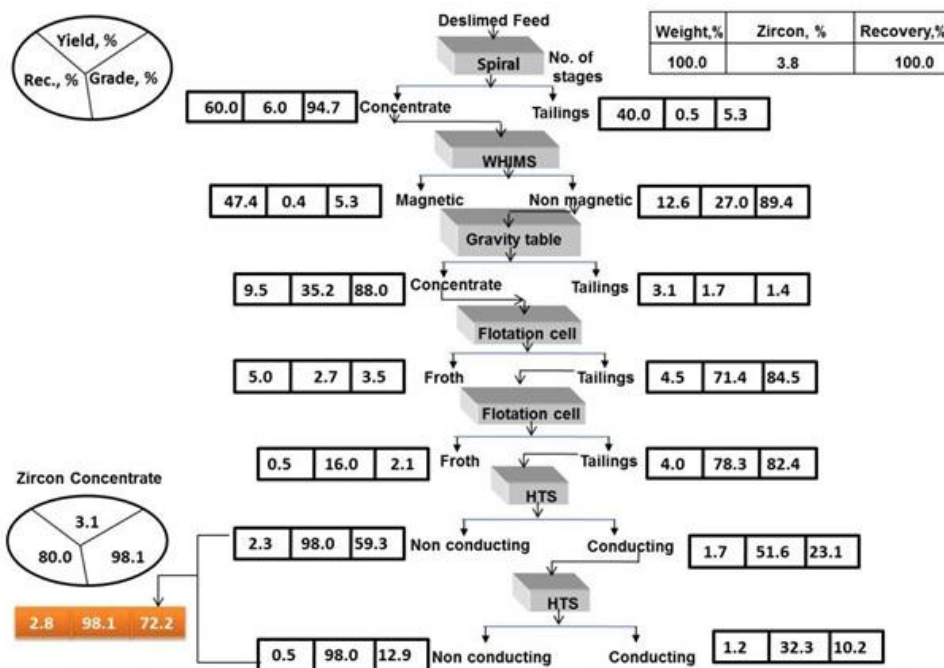


Fig 10. Flow sheet with material balance on recovery of zircon from deslimed red sediments sample of Odisha coast

The data indicate that zircon concentrate obtained by this physical separation processes contain 98.1% zircon mineral concentrate with 80% recovery from a feed sample contain 3.8% zircon mineral. The complete chemical analysis of ilmenite and zircon are shown in Tables 1 and 2. The data indicate that ilmenite mineral concentrate contain 49.1% TiO_2 , 14.98% Fe_2O_3 and 33.18% FeO . The zircon mineral concentrate contain 63.3% ZrO_2 and 31.3% SiO_2 .

Table 1: Complete chemical analysis of Ilmenite mineral concentrate [14-15]

TiO_2 %	FeO %	Fe_2O_3 %	Al_2O_3 %	SiO_2 %	Th ppm	U ppm
49.10	33.18	14.98	0.52	0.48	35	< 4

Table 2: Complete chemical analysis of zircon mineral concentrate [14-16]

ZrO_2 %	SiO_2 %	HfO_2 %	Al_2O_3 %	TiO_2 %	FeO %	Fe_2O_3 %
62.70	29.7	0.9	4.3	0.1	0.4	0.8

3.3 Value Addition

3.3.1 Ilmenite

The titanium dioxide is prepared from chemical process as well as pyrometallurgical route including conventional muffle furnace and microwave heating furnace. Initially calcined synthetic rutile product is prepared from red sediment ilmenite by generating soda ash roasted slag product which is subsequently leached by 6M HCl. Graphical representation of leaching kinetics of soda ash roasted slag of red sediment ilmenite for production of synthetic rutile can clearly be seen in Fig 11. The data indicate that white calcined synthetic rutile product is obtained by this process.

The complete chemical analysis of calcined synthetic rutile product is shown in Table 3. The data indicate that calcined synthetic rutile product obtained contain 97.21% TiO_2 , 1.68% Fe_2O_3 and traces of Al_2O_3 and SiO_2 . This product is suitable for industrial applications.



Fig 11. Graphical representation of leaching kinetics of soda ash roasted slag of red sediment ilmenite for production of synthetic rutile [15]

Table 3: Complete chemical analysis of calcined synthetic rutile product [15]

TiO_2 %	Fe_2O_3 %	Al_2O_3 %	SiO_2 %
97.21	1.68	0.31	0.27

Similarly, the red sediment ilmenite sample is subjected to reduction roasting followed by metallisation in the microwave heating furnace. In conventional furnace, it took more than 3 hrs at 1200°C and where as in microwave heating furnace, it took only 45 minutes at lower than 1000°C. The product obtained from the microwave heating furnace is subjected to SEM - EDAX for identification of titania slag and iron metal and its chemical analysis. This can be seen clearly in Fig. 12. The data indicate that SEM-EDAX analysis of ilmenite minerals heated in microwave furnace. Titania rich slag as shown in Fig. 12(A) contain TiO_2 83.3% and FeO 16.2% by weight. Similarly the metallic iron phase show`s TiO_2 5.4% and FeO 94.6% by weight in Fig. 12(B).

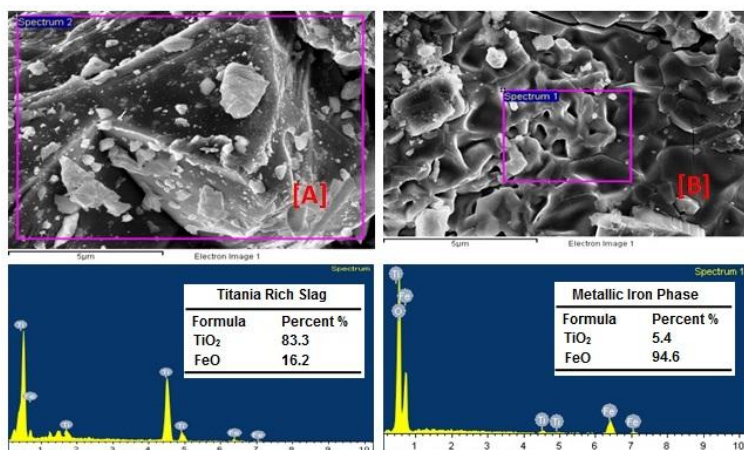


Fig 12. SEM-EDAX data of ilmenite minerals heated in microwave furnace (A) Titania rich slag and (B) Metallic iron [17]

3.3.1.1 Formation of Nano Titania particles

It is discussed in Fig.1 at materials and methods section, about the experimental procedure for preparation of nano materials from red sediment ilmenite. The composition of the dissolution liquor obtained from 500 kg batch after two stages leaching from red sediment ilmenite is given in Table. 4. The data indicate that the liquor contain TiO₂ 94.3 gpl. The hydrated titania is subjected to characterisation using Thermal analyser (TG-DTA), X-Ray Diffraction (XRD) analysis, Surface area with Brunauer–Emmett–Teller (BET) calculation and Transmission electron microscopy (TEM) analysis. The results from the TG-DTA analysis, it is found that weight loss of hydrated titania takes place up to 400°C. Maximum weight loss is found to be 97% at this temperature. XRD data for dried as well as for sample calcined at 400°C shows that hydrated titania is in anatase phase. The crystallite size (X) calculated using Scherrer’s formula and the surface area (Y) of the samples were analysed by nitrogen adsorption principle using BET principle and the results are shown in Table 5. The results of particle size (Z) carried for titania samples from transmission electron microscopy (TEM) analysis is also shown in Table 5.

Table 4: Composition of the dissolution liquor (for two stage leaching process with 96% Recovery)

TiO ₂ (gpl)	Total Fe (gpl)	FeSO ₄ (gpl)	Fe ₂ (SO ₄) ₃ (gpl)	Solid content (Kg) Un-reacted Ilmenite	Weight of TiO ₂ (Kg)
94.3	64.0	139	48.2	40	10

Table 5: Calculation for crystallite size XRD data, Surface area with BET principle and particle size with TEM analysis

Sl No	Sample	Crystallite size (X) (With XRD Data)	Surface area (Y) (With BET Data)	Particle size (Z) (With TEM Data)
1	Dried at 110 °C	4.2 nm	126 m ² /g	20 nm
2	Calcined at 400 °C	32 nm	88 m ² /g	20 to 50 nm

It is shown in Table 6 for the results of crystalline size analysis with transmission electron microscopy (TEM) analysis after Sodium Dodecyl Sulphate (SDS) addition.

Table 6: TEM analysis after addition of Sodium Dodecyl Sulphate (SDS) solution

Sample (nm)	Dried at 110 °C	Calcined at 400 °C	Calcined at 500 °C
Crystalline size	5.0	9.0	19.0

The synthesis of Nano-rutile at low temperature has been carried out by using Sodium Dodecyl Sulphate (SDS) solution. The XRD data for nano rutile is shown in Fig 13 where as transmission electron micrograph of Titania nano-particles are shown in Fig 14 with high resolution of 20 nm and 50 nm.

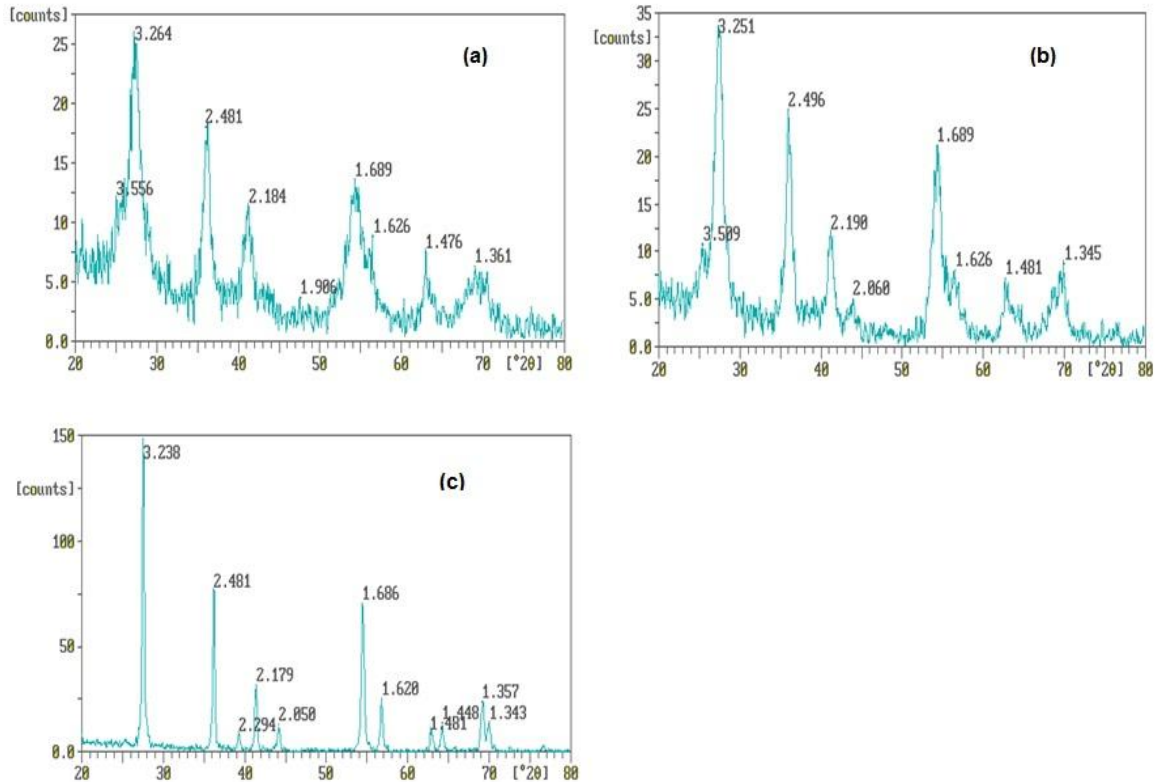


Fig 13. XRD data for nano titanium dioxide at different calcination conditions (a) TiO_2 dried at 110°C / 1h (b) TiO_2 calcined at 400°C (c) TiO_2 dried at 750°C / 2h

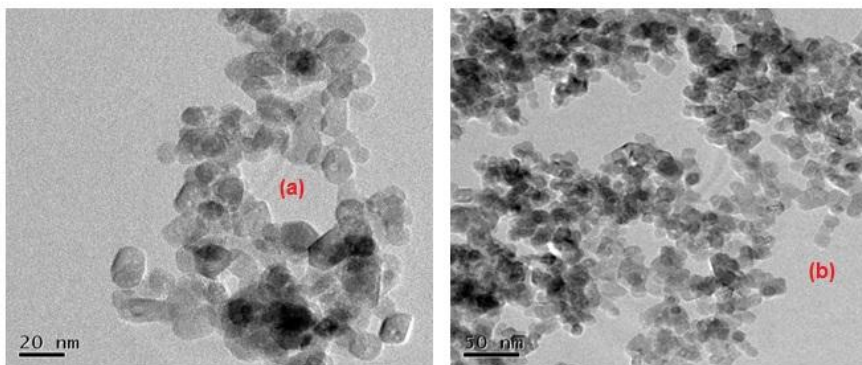


Fig 14. Transmission electron micrograph (TEM) of Titania nano-particles with high resolution of (a) 20 nm and (b) 50 nm

3.2.2 Zircon

Zircon mineral concentrate obtained from sediment sample (Heavy mineral concentrate contain 1- 4 % by weight) is ground using ball mill, agitated mill and dual planetary ball mill [16-17] for obtaining particle below $45\ \mu\text{m}$ for preparation of nano materials from zircon mineral concentrate. It is observed from the grindability characteristic of zircon that it is very difficult to get finer size and more over the process is energy intensive. The data indicate that around 6-8 hrs required to grind zircon to below $45\ \mu\text{m}$ using dual planetary ball mill. Hence natural zircon mineral obtained from red sediment is used for preparation of nano zircon.

The experimental procedure for preparation of zircon nano materials from red sediment materials, in materials and methods section is shown earlier in Fig 2. The XRD pattern for zirconium oxide nano particle is shown in Fig 15 where as XRD patterns for 3, 5, 8 mol% Ytria doped zirconium oxide shown in Fig 16. High resolution transmission electron micrograph of zirconia nano particles are shown in Fig 17. The summary of the characterisation results of zircon nano materials is shown in Table 7.

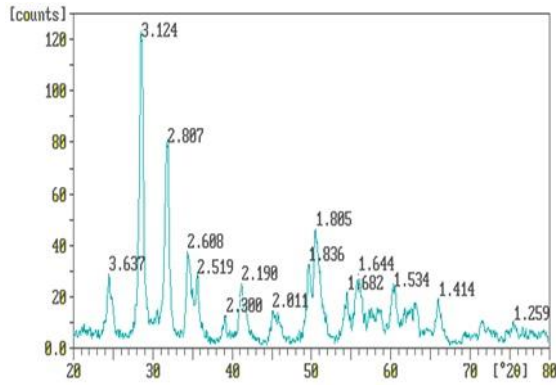


Fig 15. XRD patterns for zirconium oxide nano particle

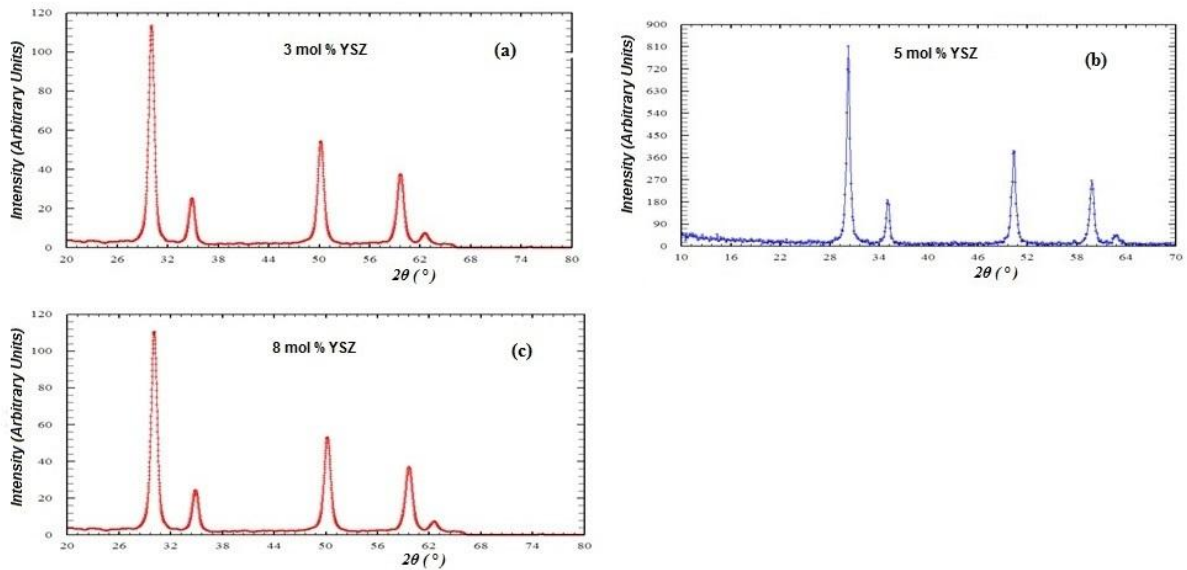


Fig 16. XRD patterns of Ytria doped zirconium oxide (YZZ) for (a) 3 mol % (b) 5 mol % (c) 8 mol %

Table 7: Summary of the characterization results of zircon nano materials

Sl No	Sample	Average particle size from TEM (nm)	BET Surface area (m ² /g)
1	Pure Zirconia	~ 30 nm	20.3
2	3 mol % YSZ	~ 20 nm	20.6
3	5 mol % YSZ	~ 20 nm	23.9
4	8 mol % YSZ	< 20 nm	24.9

The existence of nano particles has been confirmed by measuring the particle size using high resolution transmission electron micrographs which reveals the nano nature of the powder. The average sizes of the particles are found to be ~50 nm (Fig 17). The average specific surface area measured by BET method is found to be in the range of 20-24 m²/g.

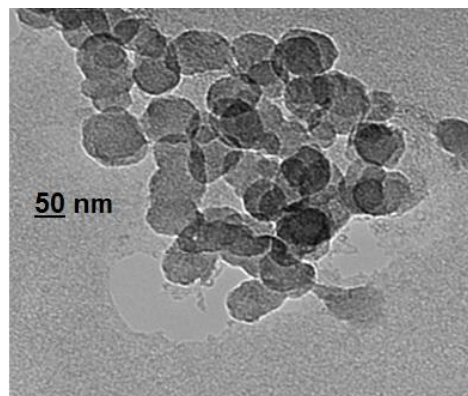


Fig 17. Transmission electron micrograph of zirconia nano particles

4. Conclusions

Indian coast possess a huge amount of heavy placer minerals. The Indian Rare Earths Limited (Government of India) and private companies are recovering individual minerals. But so far no attempt has been made to recover individual placer nano-minerals and their value addition. This paper deals with mines to nano materials from badlands topography of South East Coast of India. Following are the findings and outcomes for this research work:

- a) The badlands occur throughout the east coast of India. During rainy season, the placer minerals are being released and transported by rivers to the sea. These badlands contain 6% heavy minerals.
- b) Ilmenite contains 60 - 80% where as zircon mineral contains 1- 4 % in the heavy mineral concentrate.
- c) The study reveals that the heavy placer minerals which consist of ilmenite, sillimanite, zircon, monazite, rutile etc., recovered from badlands are finer than the beach sand minerals.
- d) The separation processes involved to recover heavy minerals from badlands are different from the beach sand minerals because of lower size range, ferrous surface coating on the minerals and absence of garnet mineral.
- e) The chemical analysis reveals that ilmenite contains 48 % TiO_2 and zircon contains 62.7 % ZrO_2 .
- f) The study reveals that one can obtain Titania rich slag upto 83% by heating ilmenite minerals in microwave furnace with less metallization.
- g) The studies also reveal that for metallisation by conventional process took 3 hrs at 1200 °C and with microwave heating furnace, it took 45 minutes at below 1000 °C.
- h) The grindability characters of ilmenite indicate that fineness of ilmenite increases with time and type of mill, whereas the grindability of zircon is found to be very difficult.
- i) It is also found that during preparation of nano materials, the higher the surface area of the ilmenite gives more favourable conditions, whereas zircon does not. Hence the ground ilmenite is used for preparation of nano materials and whereas for zircon, the natural recovered mineral without milling is used for preparation of nano materials.
- j) The results of these studies reveals that from this new resources, one can prepare nano titanium dioxide and nano zirconium oxide after recovering individual heavy minerals from badlands of South Eastern Coast of India and these are much suitable for industrial applications.

Acknowledgments

The authors are thankful to Board of Research in Nuclear Sciences (BRNS) for support to carry out this project. Authors are also thankful to Research Students, the Director, Council of Scientific and Industrial Research - Institute of Minerals and Materials Technology (CSIR-IMMT) and Chief Managing Director (CMD), Indian Rare Earths Limited (IREL) for giving facilities to carry out this work.

References

1. Babu, N., Vasumathi, N., Rao, R B., Recovery of Ilmenite and Other Heavy Minerals from Teri Sands (Red Sands) of Tamil Nadu, India. *Journal of Minerals and Materials Characteristics and Engg*, 2009, 8, 149-159.
2. Laxmi, T., Rao, R B., Badland topography of coastal belt red sediment deposits of India: A potential resource for industrial minerals. *Mines and Minerals Reporter*, 2010, 3, 12-18.
3. Yuan, S., Chen, W., Hu, S., Fabrication of TiO_2 nano particles / surfactant polymer complex film on glassy carbon electrode. *Material Science Engg*, 2005, 25, 479-485.
4. Zhang, L., Zhu, Y., He, Y., Li, W., Sun, H., Preparation and performances of mesoporous TiO_2 film photocatalyst supported on stainless steel. *Applied Catalysis B Environment*, 2003, 40, 287-292.
5. Mubarak, A K., Shauk, M M K., Mahir, A M., Sabia, S., Jahid, M M I., Jasim, U., Sensitization of Nanocrystalline Titanium dioxide Solar Cells using Natural Dyes: Influence of Acids Medium on Coating Formulation. *American Academic & Scholarly Research Journal*, 2012, 4(5), 1-10.
6. Ranjbar, M., Yousefi, M., Lahooti, M., Malekzadeh, A., Preparation and characterization of tetragonal zirconium oxide nano-crystals from iso-phthalic acid-zirconium (IV) nano-composite as a new precursor. *International Journal of Nanoscience and Nanotechnology*, 2012, 8, 191-196.
7. Behbahani, A., Rowshanzamir, S., Esmailifar, A., Hydrothermal synthesis of zirconia nano-particles from commercial zirconia. *Procedia Engineering*, 2012, 42, 992-1003.

8. Aysar, S K., Elias, S., Zakaria, A., Soltani, N., Structural and optical properties of zirconia nanoparticles by thermal treatment synthesis. *Journal of Nanomaterials*, 2016, Article ID 1913609.
9. Rouge, A L., Hamzaoui, H E., Capoen, B., Bernard, R., Cristini-Robbe, O., Martinelli, G., Cassagne, C., Boudebs, G., Bouazaoui, M., Bigot, L., Synthesis and nonlinear optical properties of zirconia-protected gold nanoparticles embedded in sol–gel derived silica glass. *Materials Research Express*, 2015, 2, doi:10.1088/2053-1591/2/5/055009.
10. Laxmi, T., Nishad, P., Jayadevan, K E., Rao, R B., Textural and concentration pattern of heavy minerals in red sediments of badlands topography Bhimunipatnam, Visakhapatnam Dist. India. *Journal of Mining and Metallurgy*, 2011, 47, 75-91.
11. Brian, R D., Joseph, A R., Process for manufacturing titanium dioxide, *US patent* 4288418 A, 1980.
12. Sindlinger, C J., Clayton, C C., A Corporation of Delaware. Process for preparing alkali metal zirconate from zircon. *US patent* US2962346 A, 1960.
13. Singh, A K., Pathak, L C., Roy, S K., Effect of citric acid on the synthesis of nano-crystalline yttria stabilized zirconia powders by nitrate–citrate process. *Ceramics International*, 2007, 33, 1463-1468.
14. Sunita, R., Development of flowsheet for recovery of individual heavy minerals from SE coast of India with special reference to in depth characterization of zircon minerals. *A PhD Thesis from Siksha 'O' Anusandhan University 2011*.
15. Laxmi, T., Development of flow sheet for recovery of individual heavy minerals from the badlands topography of SE coast of India and its value addition. *A PhD Thesis from Siksha 'O' Anusandhan University 2013*.
16. Srikant, S S., Jayasankar, K., Mukherjee, P S., Rao, R B., Effect of microwave heat treatment on grindability of zircon in a planetary ball mill. *AT International - Mineral Processing*, 2013, 54, 55-63.
17. Srikant, S S., Microwave processing of beach placer heavy minerals. *A PhD Thesis from Siksha 'O' Anusandhan University 2014*.