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Recovery of monazite from red sediments of badlands topography of south east coast of India and its characterization for industrial applications

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An attempt has been made in this paper to recover pure monazite, a source for thorium, from the red sediments badlands topography of Kalipalli village of Ganjam district of Odisha, India by developing an industrially viable process flow sheet from a feed sample containing monazite mineral 0.63 % by weight. Results have indicated that monazite purity of 98 % could be achieved with a recovery of 63 % by weight. Recovery of monazite can be enhanced if the spiral concentrate is further scrubbed in alkaline media prior to subjecting to mineral separation studies. In the present investigation an attempt is also made to compare the monazite occurring from red sediment sample with naturally occurring monazite from beach sands. It is found that in spite, their morphological and topographical characteristics are markedly different the monazite occurring from both sources, have chemical elemental similarity. It is also seen that the chemical states of rare earth (RE) elements in monazite of both sources are matching. Further, due to natural occurrence of finer particle size of monazite (< 150 micron) in red sediments sample, the material is found to be suitable for chemical treatment to recover easily the valuable rare earths from it. Hence, it is recommended for mining of red sediments for recovery of monazite from red sediments of badlands topography as an additional resource for beach sand monazite.

[**Keywords**: Badlands, Heavy minerals, Mining, Monazite, Thorium]

Introduction

India has many potential placer mineral sand resources with large reserves of economically important heavy minerals such as ilmenite, rutile, zircon, monazite, sillimanite and garnet[1-2]. Monazite is a co-product while producing these potential minerals at a desired purity[3-4]. The state wise resources of *in situ* monazite available in beach sand placer mineral deposits along the coastal tracts of India as established by Atomic Minerals Directorate for Exploration and Research (AMD), a constitute unit of Department of Atomic Energy (DAE) is shown in Table S1[(refs. 2,5)].

However, there are many resources like sea bed off coast, inland placers, the red sands also known as Teri sand in Tamil Nadu are some of the important sources of monazite. The red sediments of badlands topography are also prominent features in eastern coast in India, which run from West Bengal state in northto Odisha coastal, south from Andhra Pradesh to Tamil Nadu[6]. Several studies on red sediment deposits have indicated that red sediment heavy placer deposits contain a considerable variation in chemical and mineral composition depending upon the location[7-8]. The average heavy mineral of red sediment deposits ranges from 10 to 50 % which could be treated as a supplementary source for monazite also.

The red sediment hills of badlands topography in Odisha are extended throughout Ganjam, Khurda and Puri district. These deposits occur in semi arid regions with heavy placer mineral concentration average varying from 40 to 50 % total heavy minerals (THM), out of which 30 to 40 % are ilmenite. The other heavy minerals sillimanite (3-5 %), zircon and rutile (1-3 %), monazite and other minerals (< 2 %) are also found to be present[9].

Although, plenty of information is available on the formation and mineral assemblage of red sediments, there is little relevant information in the literature, from a practical viewpoint, to recover the heavy placer minerals especially monazite and its characterization from these minerals or any other deposits[10-13] or with reference to the study of monazite[14-20].

At present, India is recovering monazite as by-product from beach sand minerals and huge deposits of monazite for thorium, as it has been projected around 30 % of its energy demands will be recovered from non-carbon sources by the year 2030. Presently, the country has installed capacity to generate 5.78-giga watt nuclear power, where uranium has been used for nuclear power generation in power plants. These uranium-based nuclear reactors will be replaced by thorium-based nuclear power reactors and the demand of thorium[21-22] would be increased in future. Since, monazite is considered as the principal source of rare earths and thorium, the objective of the present study is to recover monazite from the red sediments of badlands topography of Odisha. The placer minerals releases from the red sediments seasonally due to tropical or cyclonic rains and these minerals are naturally very fine and partially weathered minerals[23-25]. It is also found that since these placer minerals are embedded in red sediments most of these minerals are ferrous coated. The recovery process for selective mineral is totally different from the existing beach sand mineral separation plants. In view of this, in the present paper an attempt is made to assess the feasibility to mine red sediments of badlands topography to recover pure monazite from typical badlands topography of Kallipalli village, South East Coast of Odisha, India for further studies specially for strategic applications.

Materials and Methods

Red sediment samples collected from typical badlands topography of Kalipalli village (Fig. S1) of Ganjam district of Odisha state, India (Lat 19°21’ N and Long 85°03’ E) was used for the present study. A typical red sediments deposit of Kalipalli village as shown in Figure S1(a) while Figure S1(b) shows the typical dendritic structure in the red sediments and the downstream flow of water containing fluvial heavy placer deposit concentrations. A total of 32 samples were collected in a grid pattern from these areas and mixed thoroughly to get a composite and representative sample. Typical sample collection from the field is shown in Figure S2. It can clearly be seen that the samples were collected from both the sides of walls as well as along the fluvial zone. All samples are numbered 1-32 and shown clearly inside the circles as can be seen in Figure S2.

The bulk sample was subjected to scrubbing and de-sliming to recover heavy minerals. De-sliming of the samples was achieved by using Mozley hydro-cyclone separator. Spiral concentration was carried out for the pre concentration of heavy minerals from this bulk sample using a laboratory model spiral concentrator (Model: SC20-VC), which was supplied by M/s. MultiTech (P) Ltd., to recover total heavy minerals (> 98 % grade).

De-slimed/ spiral concentrate samples were subjected to coning and quartering to get a representative sample for characterization. By using Dry High Intensity Magnetic Separator (DHIMS), Total Magnetic Mineral (TMM) and Total Nonmagnetic Minerals (TNM) percentages are calculated. Heavy liquid separation studies were carried out using bromoform (specific gravity 2.89 g/cm^3) for determining the total heavy minerals present inred sediment samples. Methylene iodide (di-iodine methane, CH_2I_2) having specific gravity of 3.3 g/cm^3 was used to determine the placer heavy minerals.

The bulk sample was subjected to a series of separation techniques such as gravity concentration, electrical and magnetic separations to produce a desired purity of monazite. Minerological modal analysis was also carried out using a Leica petrological optical microscope. Ground samples were subjected to X ray diffraction (XRD) using PANalytical (X-PERT) powdered diffractometer using Cu-Kα radiation to identify the mineral phase. Details XRD device parameters are given below.

Diffractometer type:	PANalytical X-PERT
Model:	Philips PW3710
Type of radiation:	X-Ray
Wavelength source:	Cu-Kα, λ = 0.709 Ǻ
Power:	40 KV, 20 mA
Detecting instrument:	Scintillation counter
Profile breadth:	0.04 (1)º 2θ, 0 - 65º
Specimen motion:	Stationary
Intensity measurement:	Peak height

Results and Discussion

As shown in Figure 1, the mineralogical modal analysis of the sample contains mainly ilmenite (26.35 %), followed by sillimanite (5.17 %), rutile (1.22 %), zircon (1.21 %), monazite (0.63 %), garnet (0.23 %) and rest heavy and gangue minerals (65.19 %). As clearly observed from Figure 2, the XRD data confirms that the sample contains ilmenite followed by other minerals such as sillimanite, rutile, zircon and monazite. Physical analysis of red sediment sample as shown in Table S2, indicates that the sample contains 35.29 % of THM, out of which VHM (specific gravity > 3.3 g/cm^3) are 29.13 % and LHM (specific gravity > 2.86 g/cm^3 and < 3.3 g/cm^3) such as sillimanite and other pyroboles are 5.43 % by weight. The total magnetic heavy minerals present in the sample are found to be 27.31 % measured by mineral counting method, respectively.

The size distribution of de-slimed feed (i.e a feed to spiral concentrators) sample along with the modal analysis of each size fraction is shown in Table S3. The modal analysis data as shown in Table S3, indicate that the quartz mineral is distributed maximum in the spiral concentrate in the size range between -420+250 µm. The total heavy mineral distributed maximum in the range of -180+106 µm. Further the modal analysis indicated that monazite present in the feed sample is finer and is in the range of -250+106 µm.

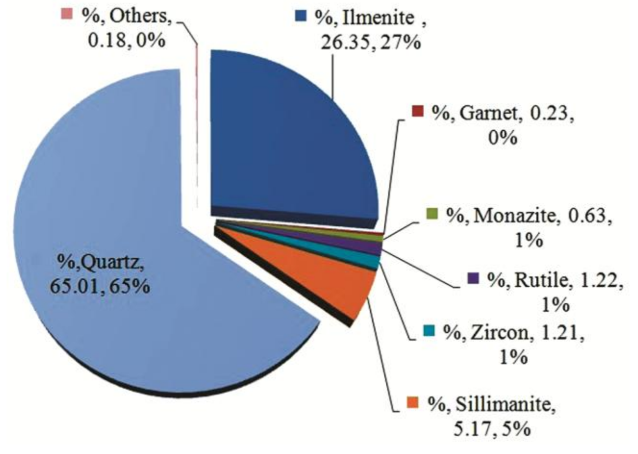

Fig. 1 — Mineralogical modal analysis of Kalipalli red sediments

Development of flow sheet on recovery of monazite

The flow sheet for recovery of monazite with material balance and other heavy minerals is shown in Figure 3. The data indicate that the spiral concentrate obtained subjected to RED contains 97.3 % THM. The magnetic product obtained from RED is found to be mostly titanium bearing minerals specially ilmenite containing 71.6 % by weight. The non-magnetic fraction contains monazite, zircon and sillimanite (28.4 % by weight). The non-magnetic fraction is further subjected to high tension separator (HTS) where conducting and non-conducting minerals are separated from the non-magnetic minerals. The conducting minerals are mostly titanium bearing minerals accounting 5.2 % by weight. The non-conducting minerals in which monazite mineral is one of them contains 23.2 % by weight. Since monazite mineral possess feebly magnetic character, which is present in the non-conducting fraction, was further subjected to Isotope-ratio mass spectrometry (IRMS). The non-magnetic fraction containing 21.2 % by weight was preserved for further recovery of other heavy minerals of commercial value. The magnetic fraction mostly contains monazite containing 2 % by weight. It is observed that the minerals present in the magnetic product shows ferrous coating on the

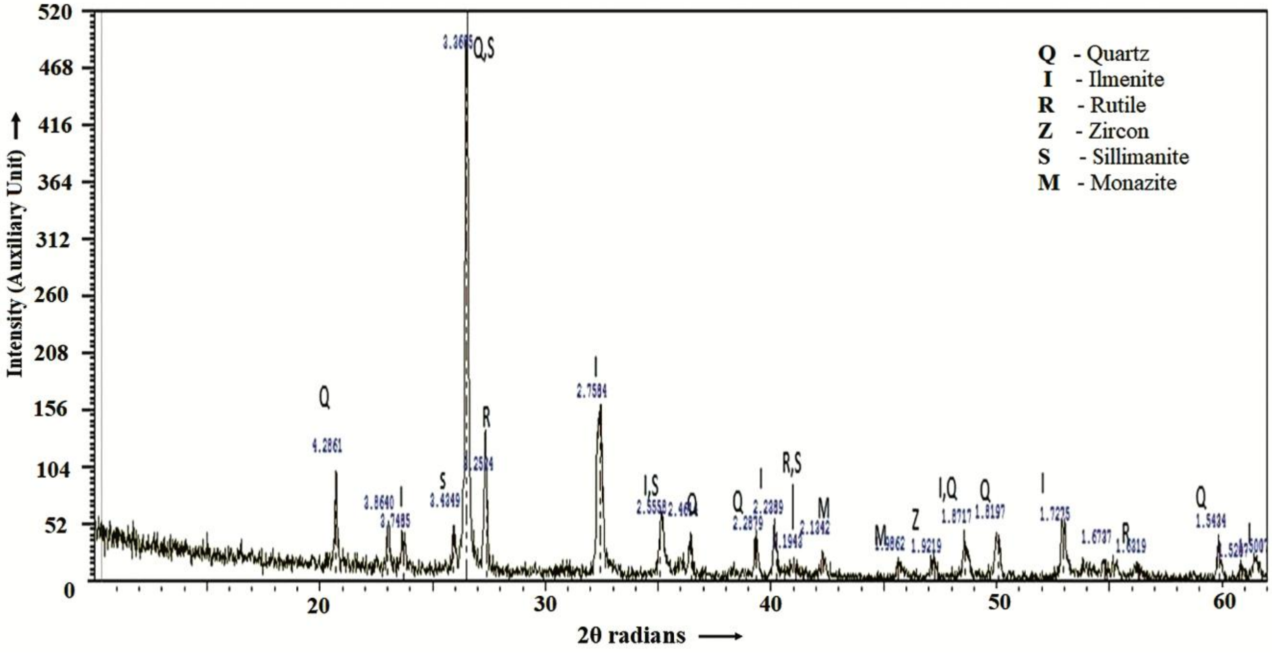

Fig. 2 — XRD pattern of de-slimed feed sample

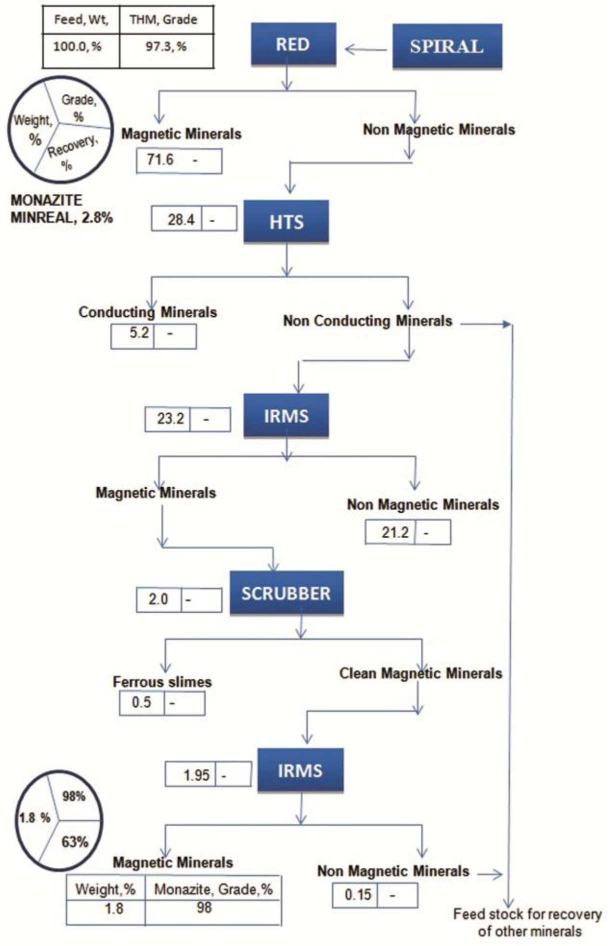

Fig. 3 — Flow sheet on recovery and material balance for monazite

individual minerals. In view of this, the magnetic minerals are further subjected to scrubbing in caustic solution. The scrubbed product was deslimed and the slimes were separated.

The deslimed product containing clean and pure magnetic minerals accounting to 1.95 % by weight is dried at 100 ºC for further separation using Isotope-ratio mass spectrometry (IRMS). The Non magnetic mineral fraction contains 0.15 % by weight which is kept for recovery other minerals of commercial value. The total magnetic minerals recovered at two different unit operations are accounting to total 21.35 % by weight. The magnetic fraction recovered from Isotope-ratio Mass Spectrometry (IRMS) after scrubbing of slimes containing 1.8 % by weight. It is found by mineral grain counting method that the grade of this magnetic fraction contains 98 % monazite. It is observed from the material balance flow sheet (Fig. 3) that the spiral concentrate contains around 2.8 % monazite mineral content and the product obtained contain 1.8 % by weight with 98 % grade and the recovery of monazite from this sample is around 63 % by weight.

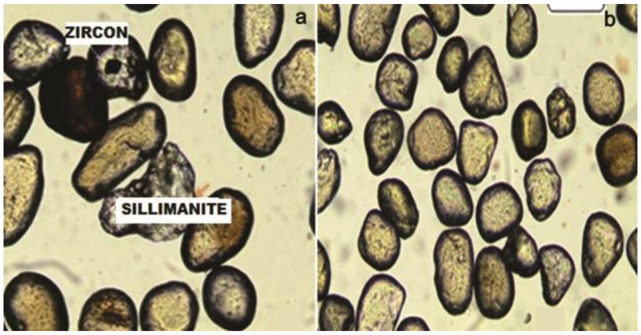

Fig. 4 — (a) Optical monazite image purity of 96.5 % and (b) 98 % purity

Table 1 — Size analysis of feed and Monazite product

Size (µm)	Spiral feed (wt %)	Monazite product (wt %)
-300	23.0	0.0
- 300 + 250	28.3	0.0
- 250 +180	13.2	1.6
- 180 +150	21.5	38.1
- 150 +106	12.3	49.2
-106	1.7	11.1
Total	100.0	100.0

Further, an attempt was made to produce different grades of monazite ranging from 92 to 98 % and the correlation between ‘recovery and purity of monazite’ is shown in Figure S3. From the figure, it may be seen that recovery of monazite mineral decreases with increase in grade. Typical micro photographs of monazite minerals obtained different process containing 96 % and 98 % grade samples are shown in Figure 4. The data indicate that there are other minerals like sillimanite etc. are present in the 96 % grade of monazite.

Characterization

The particle size analysis of spiral feed i.e de-slimed feed, monazite mineral present in the spiral feed, the end product containing rich in monazite and monazite mineral and the distribution of monazite mineral in the end product are shown in Figure S4. The data indicate that the spiral feed is very coarse due to presence of coarse quartz minerals. The size distribution of monazite in spiral feed as well as in the end product is almost similar, which indicates that the monazite was recovered with the designed flow sheet. It can clearly be seen from the size analysis of spiral feed and monazite product given in Table 1 indicate that the spiral feed is very coarse where 65 % quartz mineral was present (Figure 1-Modal analysis data) and whereas the monazite mineral remains only below the size range of 180 µm. Further it is also explained earlier that the red sediment placer minerals are well coated with ferrous materials. Hence removal of entire ferrous slimes from total heavy minerals takes more time and expenditure. In view of this, in the present investigation, only the pre concentrate of monazite is scrubbed and subjected to IRMS for recovery of high grade of monazite. The recovery of monazite could be improved if the entire THM is scrubbed with alkali media and subjected to further processing which is difficult at present stage. The main aim of the present study is to recover pure monazite and its observation for strategic applications.

A comparison of monazite obtained from the red sediment source with other sources such as Chavara (Kerala), MK (Tamil Nadu) and Chatrapur (Odisha) is presented in Table S4. Table S5 shows the comparison of Bragg d value for the 98 % pure monazite fraction obtained from red sediment with that of monazite sourced from beach sand of Chatrapur, Odisha deposit (for reference) and Joint Committee for Powder Diffraction System (JCPDS) values. Figure 5 shows the XRD pattern of 96.5 % pure monazite and 98 % monazite recovered from the present sample. Figures 6 and 7 show the Energy-dispersive X-ray spectroscopy (EDS) spectra recorded at 500X magnification for both Beach sand (OR) monazite pellets and Red sediment (RS) monazite pellets respectively.

The Energy-dispersive X-ray spectroscopy (EDS) X-ray mapping sum spectra for Red sediment (RS) monazite and beach sand (OR) monazite powders are shown in Figures S5(a and b), respectively. The EDS results show uniform distribution of elements in both samples. The elemental composition obtained by EDS analysis asshown in Table 2, indicate that the rare earth elements present in both the samples contain almost similar distribution.

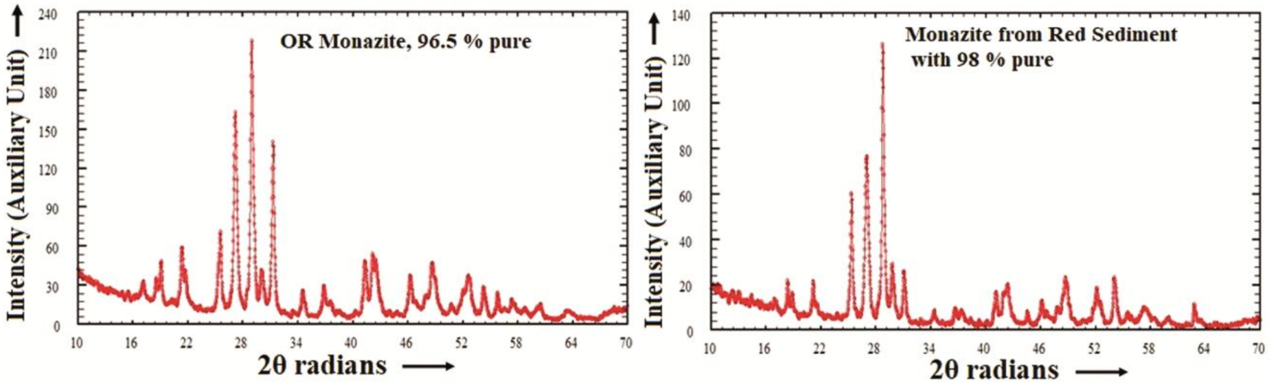

Fig. 5 — XRD pattern of Monazite from: (a) beach sand and (b) red sediments

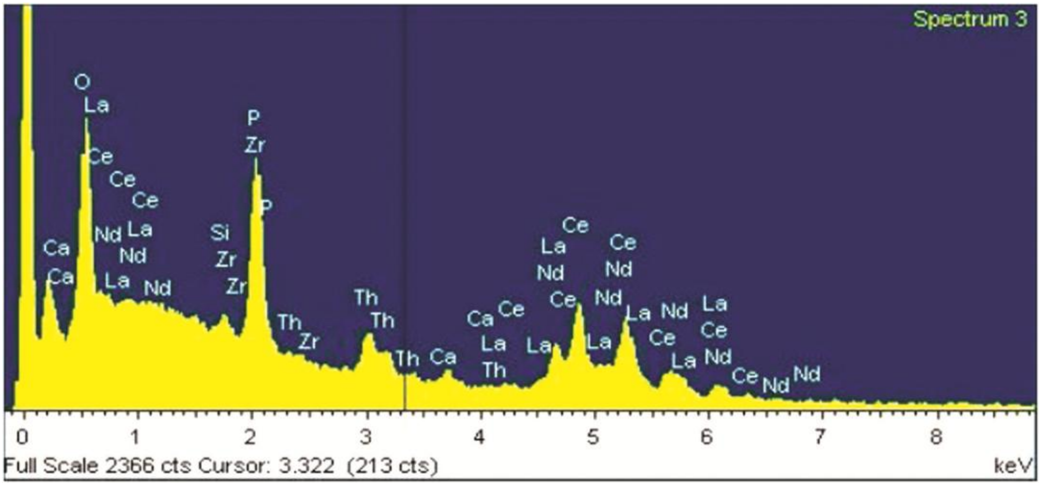

Fig. 6 — EDS spectra for Beach sand (OR) monazite pellet at 500X magnification

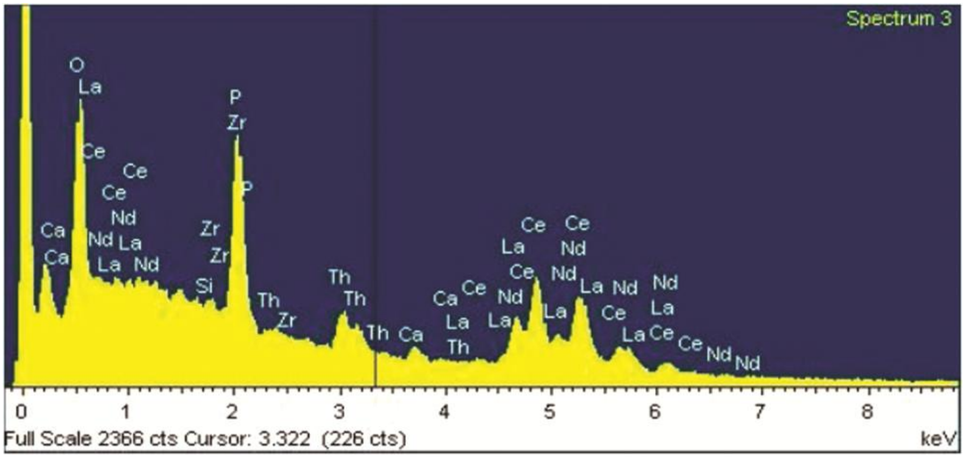

Fig. 7 — EDS spectra for Red Sediment (RS) monazite pellet at 500X magnification

Table 2 — Energy-dispersive X-ray spectroscopy (EDS) elemental analysis

Element	RS Monazite		OR Monazite	
	Range (wt %)	Average (wt %)	Range (wt %)	Average (wt %)
O	33.92-35.41	34.61	34.53-36.1	35.12
Si	0.54-1.21	0.93	1.18-2.22	1.53
P	10.57-12.06	11.28	10.63-11.33	10.96
Ca	0.92-1.1	0.99	0.71-0.87	0.82
Zr	1.45-3.64	2.90	3.74-4.56	4.16
La	10.32-11.5	10.91	9.75-9.96	9.81
Ce	22.28-24.24	23.19	21.4-21.81	21.64
Nd	7.69-8.64	8.03	7.22-8.09	7.66
Th	6.65-7.76	7.12	7.21-8.71	8.28

The X-ray photoelectron spectroscopy (XPS) survey scan and its high-resolution spectra of Red sediment (RS) monazite sample is shown in Figures S6(a and b), respectively. The binding energy (B.E) values of $La_{3d5/2}$ and $La_{3d3/2}$ peaks suggest presence of La^{+3}, likewise $Ce_{3d5/2}$ and $Ce_{3d3/2}$ peaks suggest presence of Ce^{+3} in the RS monazite sample. Similarly the X-ray photoelectron spectroscopy (XPS) survey scan along with its high resolution spectra of beach sand (OR) monazite sample is shown presented in Figures S7(a and b), respectively. The binding energy values of $La_{3d5/2}$ and $La_{3d3/2}$ peaks suggest presence of La^{+3}, likewise $Ce_{3d5/2}$ and $Ce_{3d3/2}$ peaks suggest presence of Ce^{+3} in the OSCOM monazite sample (OR). Figures 8(a and b) shows the scanning electron microscopy (SEM) analysis at a magnification of 15000X forred sediment (RS) and beach sand (OR) monazite samples respectively. Table 3 summarizes the morphological and topographical characteristics.

The results obtained from above analysis shows that there exist significant differences in properties between monazite product of coastal and red sediments of badlands topography. It is observed that the particle size distribution of coastal monazite is coarser than that of red sediment origin. Similarly, the thick ferruginous coating present on monazite of red sediment is also more pronounced as compared to coastal monazite. The concentration of monazite in heavy minerals recovered from red sediments is found to be higher than that of heavy minerals recovered from Chatrapur beach sand deposit although both the sources are not far off from each other. As seen in XRD and EDS analysis, monazite from both sources, i.e., red sediment and beach sand placers has chemical elemental similarity. Also, the chemical states of monazite of both sources are matching as per the XPS analysis. However, their morphological and topographical characteristics are markedly different as per SEM analysis.

Hence, mining of red sediments of badlands topography is also one of the options because it has the potential resources for placer industrial minerals and specially monazite, which is the main source for thorium and important for the country's third stage nuclear power sector and has also the potential to emerge as next generation fuel for nuclear power plants.

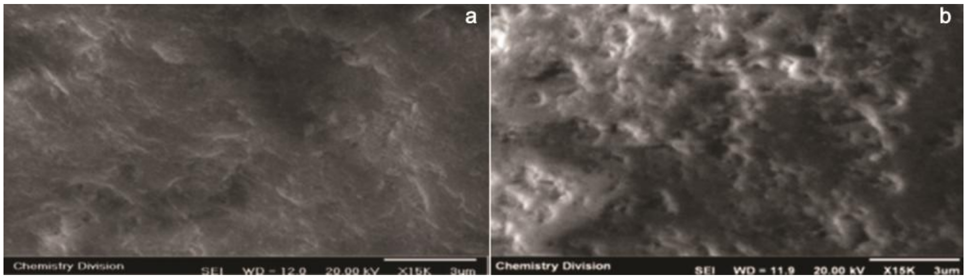

Fig. 8 — SEM analysis at 15000X magnification for: (a) Red sediment (RS) and (b) Beach sand (OR) monazite

Table 3 — Summary of SEM morphological and topographical analysis

Samples	RS Monazite	OR Monazite
Nature	Non-conducting	Non-conducting
Particle size	60-290 µm	140-620 µm
Shape	Oval	Oval
Length: Width	1.6 - 1.16:1	1.7- 1.14:1
Topography	Particle surface is wrinkled, mainly have sharp edges and several small particles (size 150-500 nm) are seen all over the surface	Surface features mainly have round edges and several pore (200-800 nm) like openings

Conclusion

The beneficiations, mineralogical, physical and chemical characteristics of monazite, obtained from the red sediments of badlands topography of Kalipalli village of Ganjam district of Odisha, India are carefully studied in the present investigation. It contains 0.63 % of monazite mineral by weight. This monazite which is recovered by using gravity spirals, followed by dry high intensity magnetic separators and high-tension separators contain 98 % purity of monazite with 63 % recovery which could be improved by scrubbing the spiral feed in alkali media prior to mineral separation. Yield of monazite could also further be increased by recovering monazite from other circuits of flow sheet by integration and recycling process. In the present investigation, an attempt is also made to compare the monazite occurred from red sediment sample with naturally occurring monazite from beach sands. It is found that in spite, their morphological and topographical characteristics are markedly different but the monazite occurred from both sources has chemical elemental similarity. It is also observed that the chemical states of rare earth (RE) elements in monazite of both sources are matching. As seen in XRD and EDS analysis, monazite from both sources, i.e., red sediment and beach sand placers has chemical elemental similarity. Also, the chemical states of rare earth (RE) elements in monazite of both sources are matching as per the XPS analysis. However, their morphological and topographical characteristics are markedly different as per SEM analysis. It is observed that the monazite occurred from red sediment sample is finer than the beach sand sample. The finer size of monazite (< 150 micron) makes it suitable for direct digestion with caustic lye instead of pulverizing and digesting with lye.

Hence, mining of red sediments of badlands topography is also one of the option as an addition resource because it is potential resources for placer industrial minerals and containing specially monazite, which is the main source for thorium and important for the country's third stage nuclear power programme and has the potential to emerge as next generation fuel for nuclear power plants.

Supplementary Data

Supplementary data associated with this article is available in the electronic form at http://nopr.niscair.res.in/jinfo/ijms/IJMS_49(09)1528-1535_SupplData.pdf

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Conflict of Interest

The authors have No conflict of interest to declare.

Author contributions

CKA wrote the manuscript with support of NM, RBR & SSS. Both CKA & RBR did sample collection and its preservation, mineralogical characterization & analysis with the help of SSS. CKA: contributed under the supervision of NM & RBR. SSS, RBR & NM: Identifying, testing and main analysis. Monazite recovery from badlands has not been attempted so far, elsewhere.

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