

Article

Distribution, Separation and Characterisation of Valuable Heavy Minerals from the Brahmaputra River Basin, Kurigram District, Bangladesh

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Abstract: Bangladesh's heavy minerals deposits in river systems remain largely unexplored with past research restricted to beach placer deposits along the south-eastern coastline of the country. In this study, 64 samples were collected from stable sand bars from the northern Brahmaputra River. The average total heavy minerals (THM) from all sample sites was 10.73 wt%, with the THM concentrations generally highest at the outermost edges of the river and lowest within the mid-channel regions. There was no variation in THM content from north (upstream) to south (downstream). Valuable heavy minerals (VHMs) magnetite, ilmenite, garnet and zircon made up around 25–30 wt% of the THM content, with other heavy minerals including amphibole, pyroxene, kyanite, sillimanite, monazite, apatite and xenotime making up the remaining 70–75 wt%. A VHM distribution map showed that the upstream division of the studied area appeared to be the most prospective for valuable heavy minerals. A bench scale heavy mineral sands beneficiation flowsheet involving unit processes employing gravity, magnetic and electrostatic properties was designed and based on a detailed characterisation and phase analysis of the resulting fractions, it was determined that VHMs made up ~2 wt% of the material recovered. Of these, garnet and ilmenite made up the bulk of the VHMs at levels of 0.88 and 0.51 wt%, respectively, with recoveries of >83%. Characterisation of the concentrates showed that further refinement of the processing conditions is required to generate concentrates that are potentially suitable for commercial applications.

Keywords: heavy minerals; distribution; separation; characterisation; Brahmaputra River sands



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1. Introduction

Heavy mineral (HM) sands are occurrences of high specific gravity ($SG > 2.9$) detrital minerals in siliclastic sediments that include minerals such as rutile, zircon, ilmenite, magnetite, sillimanite, monazite, chromite, tin, tourmaline, garnet and staurolite. Some heavy minerals are of more economic value than others due to their usefulness in industrial products such as pigments (ilmenite, rutile and leucoxene), ceramics (zircon) or for the recovery of high-value components such as rare earth oxides (monazite). Other heavy minerals, of lesser value, such as garnet, magnetite and tin are also potentially recoverable for commercial applications.

Bangladesh has HM sands in beach and river system placer deposits (Figure 1). Beach sand occurrences are prevalent along the southern and south-eastern coast lines (Cox's Bazar, Teknaf and Kuakata beaches) and in offshore islands (Saint Martin's, Kutubdia, Nijhum Dweep and Maheshkhali islands) in the Bay of Bengal [1–3]. On average, the beach

sand HM deposits contain 20–25 wt% total heavy minerals (THM) with mineralogical analysis indicating an average percentage of economically important valuable heavy minerals (VHM) such as zircon (4.2 wt%), rutile (2.0 wt%), ilmenite (26.0 wt%), garnet (6.4 wt%), kyanite (3.9 wt%), leucoxene (2.3 wt%), magnetite (1.9 wt%) and monazite (0.3 wt%) [4,5]. While the coastal sand deposits are high-grade, opportunities are limited with deposits located in populated, environmentally sensitive areas. In addition, some of the contained VHMs such as zircon and monazite have high levels of radioactivity [6–9]. Both make exploitation problematic.

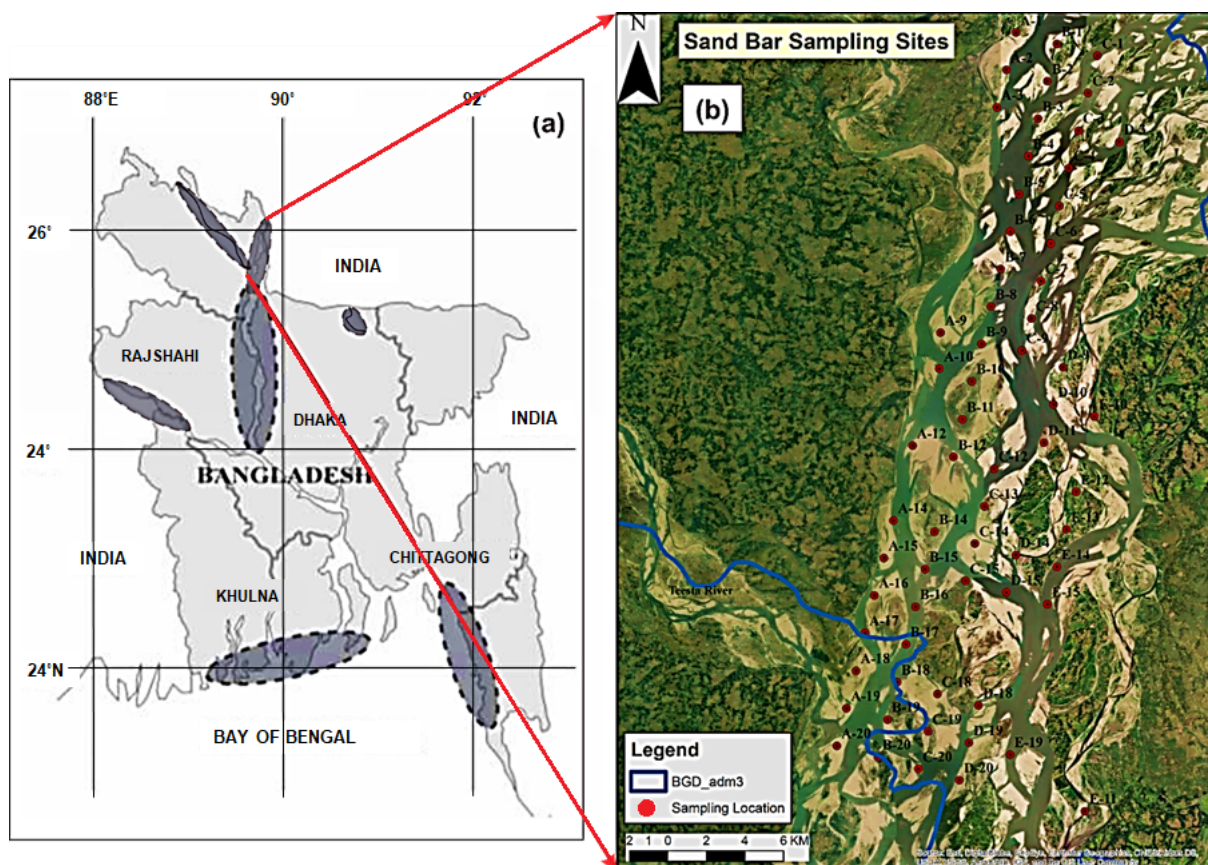


Figure 1. Location maps showing (a) the distribution of significant concentrations of heavy mineral sands in Bangladesh and, (b) sand bar sampling sites (red dots) along a 40 km stretch of the northern Brahmaputra River, Kurigram District (blue line shows the Kurigram District border).

The occurrence of large amounts of HMs in the bedload of Brahmaputra-Jamuna River sediments has been known for decades [10–15]. Recent studies to examine the potential for recovering VHM from sand bars of the northern Brahmaputra-Jamuna River have been reported by Rahman et al. [16–18] and Khalil et al. [19]. Rahman et al. [18] determined that the THM percentage of the Brahmaputra-Jamuna sand bars was lower than the beach sand deposits, ranging from 4–17 wt%. The HM assemblage comprised amphibole, epidote, garnet, pyroxenes and kyanite along with minor amounts of VHM components ilmenite (4.7 wt%), magnetite (4.4 wt%), titanomagnetite (0.9 wt%), titanite (0.9 wt%) and rutile (<0.1 wt%). Although lower grade, the uncertainty around future mineral sands exploitation from beach sands means there is emerging potential for HM sands mining and processing from river sand deposits in Bangladesh. While exploitation of VHMs from river sand deposits is uncommon, primarily due to the typically low grades encountered, the river systems in Bangladesh are constantly renewed through annual transport of ~1800 tons/km² of sediment resulting in an annual discharge of suspended

load of 540–1157 million tons/year [14]. The high annual sediment influx creates social and environmental hazards in river systems such as the Brahmaputra which is used as a key transport corridor. Annual dredging is therefore required to maintain open waterways and capturing and recovering valuable HMs from waste dredge material may potentially provide a cost-effective long-term source of VHMs—provided the recovered materials meet market specifications.

Recently, the authors carried out mineralogical characterisation studies on northern Brahmaputra River sand deposits [18]. The sediments were found to be low in clay content and the HMs were found to be well liberated and angular to sub-rounded in texture and mostly in the $-125 + 63 \mu\text{m}$ size range. This size range is optimal for VHM recovery via traditional mineral sands processing operations. Rahman et al. [18] also conducted preliminary beneficiation studies that exploited the magnetic properties and compositional variation within the iron- and titanium-rich components (magnetite, ilmenite, rutile) to examine the feasibility of generating commercial products. The present investigation extends these initial beneficiation investigations by conducting distribution and recovery studies on other components of the VHM suite. A bench scale HM beneficiation flowsheet utilising unit processes employing gravity, magnetic and electrostatic properties of the minerals was used to separate the individual VHM for further characterisation. Results from in-depth characterisation studies on four of the more valuable and commercially exploitable components, ilmenite, zircon, garnet and magnetite are presented.

2. Materials and Methods

2.1. Raw Materials

Samples were collected from sand bars within the northern Brahmaputra River, south of its entry point into Bangladesh and north of its confluence with the easterly flowing Tista River (Figure 1a). Sampling locations were in a grid pattern covering an area of 400 km^2 extending 40 km in length and 10 km in width. The spacing between each of the sample locations was approximately 2 km (Figure 1b). The depth of the samples was from the surface to ~3 m and ~50 kg of sample was collected from each location for the characterization and beneficiation studies. To assess potential variability in the THM and VHM assemblages within the study area, the sampling region was divided into three sectors, upstream, midstream and downstream, extending along the river from north to south. In total, 64 samples were collected for analysis.

2.2. Beneficiation

Concentration of heavy mineral sands from the primary ore utilises the differences in specific gravity (SG) and in the electrostatic and magnetic properties of the VHM. The concentration is carried out in two ways: wet, utilising sizing and gravity differentiation between THM, VHM, clay and quartz and dry, exploiting the magnetic, electrostatic and, to a lesser extent, the specific gravity properties of the minerals of interest.

2.2.1. Preliminary Float/Sink Analysis

Prior to the beneficiation study, a 10 g sub-sample from each location was initially subject to dense media gravity separation using the heavy liquid, bromoform ($\text{SG} = 2.89$), to provide an indication of the THM content at each location. Following separation, optical light microscopy-based point counting of polished THM grain mounts was used to determine the abundance of the VHM—ilmenite, zircon, rutile and garnet—in each of the samples.

2.2.2. Laboratory Scale Wet and Dry Processing

After the preliminary float/sink tests, the remaining sand sample from each location was used for the separation of the VHM fractions by applying bench scale separation techniques based on the physical properties of the minerals. A flow chart for the processing to achieve the separations is shown in Figure 2.

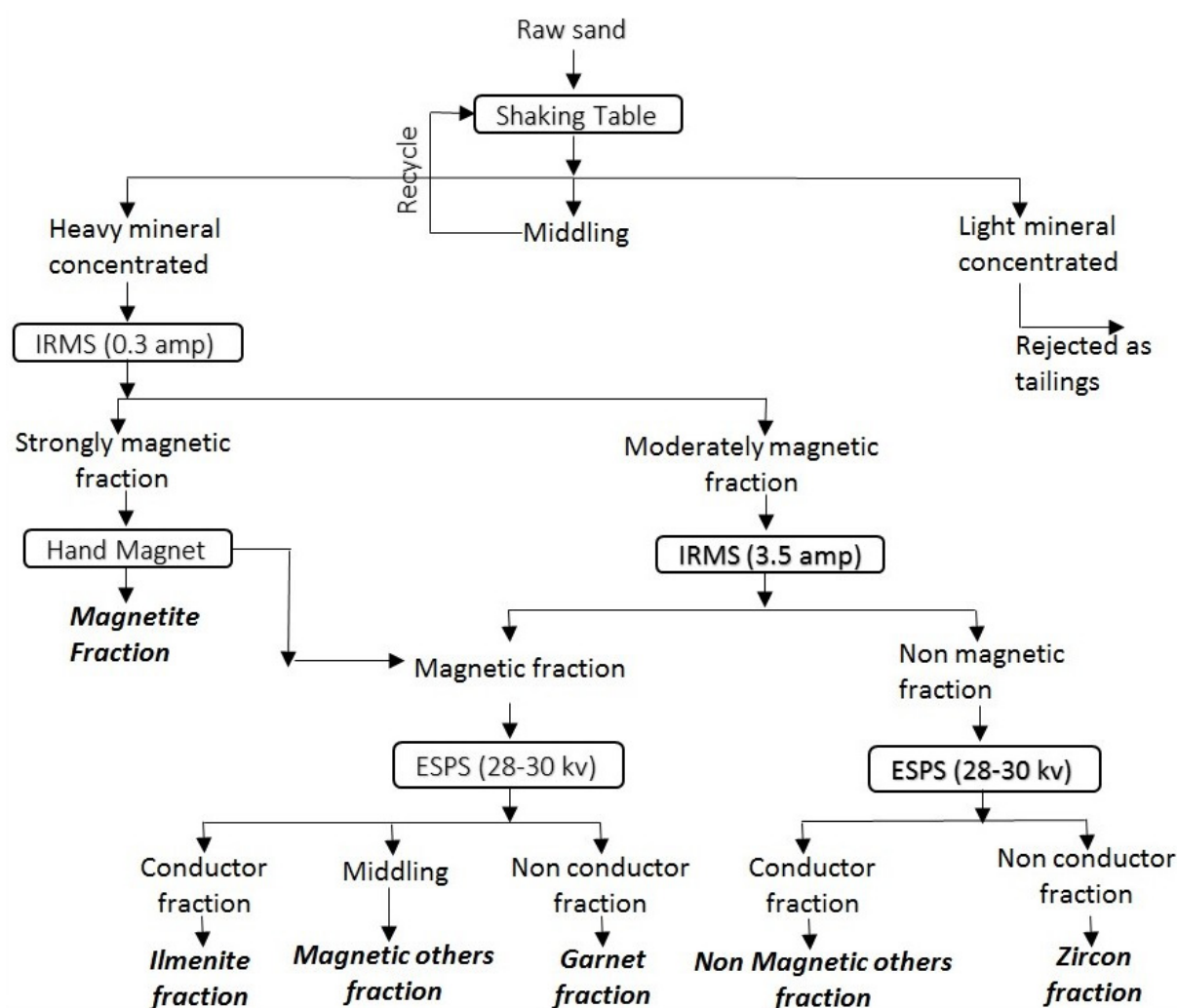


Figure 2. Flow chart showing the procedure for separation of VHM fractions from the bulk sand sample.

Initially, each sample was processed by wet gravity separation using a laboratory model Shaking Table-800, supplied by Holman-Wilfley Ltd, Redruth, Cornwall, UK. The wet separation was carried out at 30 wt% solids concentration, 12 L/min wash water and 20 kg/h throughput. This process separated each sample into a light and a heavy fraction. The light fraction contained the lower SG mineral components such as quartz, feldspar and micas and was not used in subsequent testing.

The heavy fractions were processed using an induced roll magnetic separator (IRMS—Outotec MIH (13) 111-5, Jacksonville, Florida, USA) operating at 0.3 amp and a constant rate of feed of 50 kg/h to recover the strongly magnetic heavies from the moderately magnetic heavies. Magnetite was concentrated using a hand magnet from the strongly magnetic heavies leaving behind a fraction expected to be ilmenite-rich. The moderately magnetic fraction was then further magnetically separated at 3.5 amp and at the same feed rate. Ilmenite and garnet were the main minerals in the moderately magnetic heavies and zircon and rutile were the main minerals in the non-magnetic heavies. An electrostatic plate separator (ESPS) operated at 28–30 kV was used to further separate ilmenite (conductor) and garnet (non-conductor) from the moderately magnetic heavies and zircon (non-conductor) from the non-magnetic heavies. Before electrostatic separation, the fractions were heated to about 200 °C to ensure the surfaces were free of moisture which could affect the separation.

The complete beneficiation process produced six fractions: a magnetite-rich fraction, an ilmenite-rich fraction, a garnet-rich fraction, a zircon-rich fraction, a magnetic (others) fraction and a non-magnetic (others) fraction (Figure 2). These fractions were subject to

further characterisation with emphasis placed on the two most valuable concentrates, ilmenite and zircon.

2.3. Characterisation of VHM

Chemical analysis of the beneficiated products was carried out using X-ray Fluorescence (XRF) spectroscopy while the minerals in the products were characterised using Quantitative X-ray Diffraction (QXRD), Scanning Electron Microscopy (SEM) and Electron Probe Microanalysis (EPMA).

2.3.1. X-ray Fluorescence (XRF) Spectroscopy

Chemical analysis of all six fractions was carried out using XRF. This involved milling the samples then accurately weighing ~0.4 g of the finely ground, oven dried powders into a 95% Pt/Au crucible with ~4 g (also accurately weighed) of 12:22 lithium tetraborate: metaborate flux. Each mixture was then fused into a homogeneous glass disc over an oxy-propane flame at a temperature of approximately 1050 °C and the molten material was poured into a 32 mm diameter 95% Pt/Au mould pre-heated to a similar temperature. The melt was then cooled by air jets for approximately 60 s. The resulting glass discs were analysed on a Philips PW2404 XRF system using a control program developed by Philips and algorithms developed in-house by CSIRO.

2.3.2. Quantitative X-ray Diffraction (QXRD)

Quantitative X-ray diffraction was carried on micronized samples of the magnetite-rich, ilmenite-rich, zircon-rich and garnet-rich mineral products to determine their mineralogy. Diffraction data were collected over the range 5–140° 2 θ using a Panalytical Empyrean instrument fitted with a Co X-ray tube operated at 40 keV and 40 mA. Data were collected with a nominal step size of 0.0263° 2 θ (~2 h per scan). Phase identification was performed using Panalytical Highscore Plus© software (V4.8). Quantitative phase analysis (QPA) was carried out via the Rietveld method [20] using TOPAS V6 software.

2.3.3. Scanning Electron Microscopy (SEM)

Scanning electron microscopy was used to examine the mineralogy and textures in the samples. Back-scattered electron SEM images were obtained using a FEI Quanta 400 FEG-ESEM. The instrument was operated at high vacuum (<10^{−5} Torr), an accelerating voltage of 15 keV and a beam current of 0.3 nA. The chemistry of individual phases was determined by performing spot energy dispersive analyses with data collected using a Bruker X-Flash 5010 ED detector.

2.3.4. Electron Probe Microanalysis (EPMA)

The high value ilmenite and zircon concentrates were mapped using a high resolution field emission gun equipped EPMA (JEOL 8500F Hyperprobe) to examine the distribution of major and minor mineral phases and to examine the chemical homogeneity of the grains. Polished epoxy resin grain mounts were mapped over a grid of analysis points using a combination of wavelength dispersive (WD) and energy dispersive (ED) spectroscopic techniques. The element distribution data was then processed using the software package CHIMAGE [21] which incorporates an automated clustering algorithm that identifies chemically alike phases [22–24]. Groupings of elements identified via the clustering represent statistically different chemical/mineral phases and these phases were overlaid onto the mapped region to provide a phase-patched map.

3. Results and Discussion

3.1. Distribution of THMs and VHMs

3.1.1. Total Heavy Minerals

The total heavy minerals (THM) present in the sands collected from the sample locations are listed in Table 1. The average THM content across all sample sites was 10.73 wt%

with the highest and lowest THM concentrations within the upstream, midstream and downstream regions of the sampling area as follows: 20.40 wt% to 5.40 wt%; 23.47 wt% to 3.71 wt%; and 15.90 wt% to 4.69 wt%, respectively. To assess the variability in the THM content across and along the river basin, transect plots are shown in Figure 3a,b, while a plot showing the concentration of THM's measured at all sample locations is shown in Figure 3c.

Table 1. THM contents (wt%) from the sand samples determined by float-sink analysis using bromoform. Samples were divided into upstream, midstream and downstream according to their location within the sampled area (Figure 1). Numbers in bold represent the lowest and highest THM contents within each sector.

Upstream		Midstream		Downstream	
Sample ID	THM (wt%)	Sample ID	THM (wt%)	Sample ID	THM (wt%)
A-1	5.40	A-10	10.19	A-15	4.69
A-2	12.54	A-12	23.47	A-16	6.61
A-3	9.30	A-14	10.25	A-17	9.11
B-1	6.90	B-9	9.22	A-18	10.50
B-2	11.60	B-10	15.70	A-19	12.80
B-3	10.82	B-11	11.50	A-20	15.90
B-4	8.51	B-12	10.29	B-15	9.70
B-5	20.40	C-8	11.22	B-16	10.79
B-7	9.10	C-9	3.91	B-17	12.70
C-1	7.21	C-12	7.50	B-19	15.60
C-2	9.62	C-14	3.71	B-20	8.87
C-3	17.20	D-9	10.59	C-15	12.01
C-4	5.41	D-11	11.30	C-19	13.20
C-5	5.84	D-14	15.13	C-20	5.40
C-6	15.22	E-10	20.20	D-15	8.11
C-7	11.70	E-11	10.13	D-20	10.30
-	-	E-12	9.81	E-15	14.90
-	-	E-13	8.02	E-19	12.32
-	-	E-14	7.20	-	-
Average	10.42	Average	11.02	Average	10.75

Figure 3a shows the trend of THM concentrations across the river channel from west to east, a distance of approximately 10 km in width. Results indicated that THM concentrations were highest at the outermost edges of the river and lowest within the mid-channel regions. The same pattern was observed for all three sample area divisions (upstream, midstream and downstream). It is presumed that the THM distribution reflects higher flow velocities toward the central part of the river system compared to the eastern and western margins. The distribution of THM from north to south along the channel is shown in Figure 3b. The data were taken from the mid-channel sample sites (sample locations pre-fixed by B and C) as these provided the most complete set of data. Considerable fluctuation was apparent in the data; however, the results in Figure 3b suggested there was no significant variation in THM content from north (upstream) to south (downstream). We speculate that the fluctuation in the data is likely due to the frequent shifting of stream channel and sand bars along the river, typical characteristics of a braided river system.

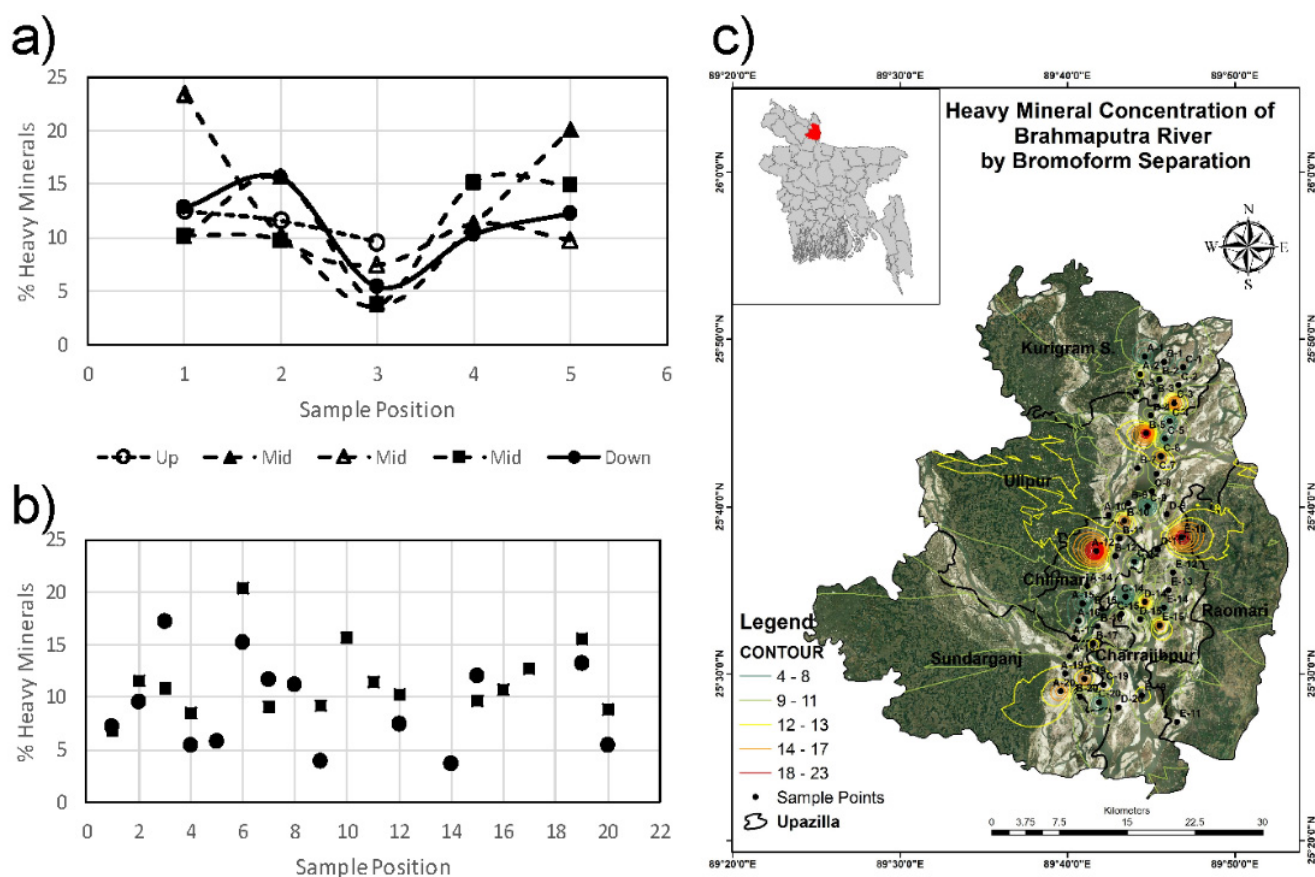


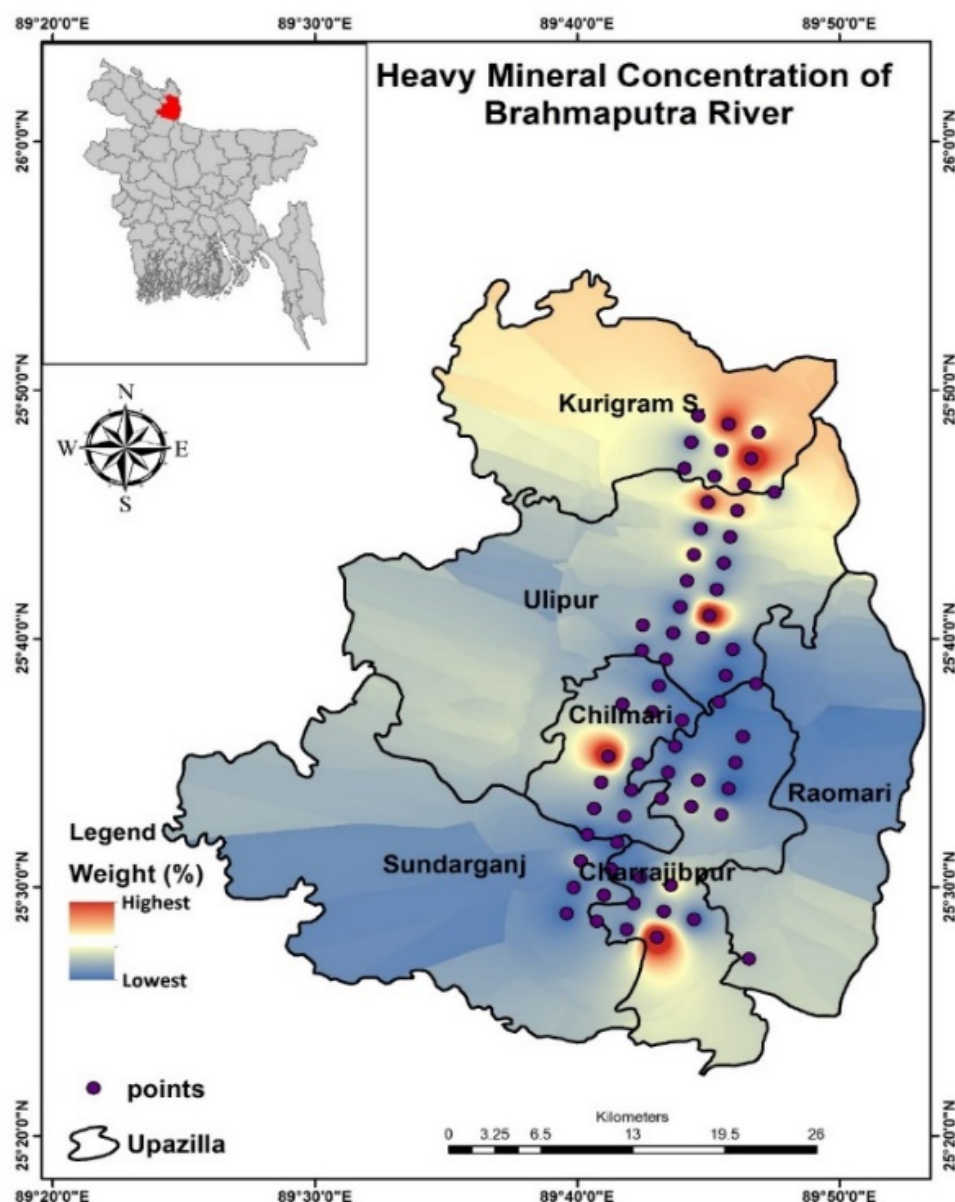
Figure 3. Plots showing the distribution of THM in Brahmaputra River samples according to location. (a) THM distribution across the river channel from west (position 1) to east (position 5) plotted according to location. Points represent data from cross-sections taken upstream, midstream and downstream. Position 1 is situated on the west bank, positions 2, 3 and 4 are mid-channel and position 5 is the east bank. (b) Distribution of THM's from north (upstream—LHS) to south (downstream—RHS). Samples are from borehole locations along the B and C transects, i.e., mid-channel locations, (c) a map showing the amount of THMs measured at each sample location.

3.1.2. Valuable Heavy Minerals

Point counting on the polished THM grain mounts was used to determine the abundance of the valuable heavy minerals; magnetite, ilmenite, rutile, zircon and garnet in the samples. Results for the average VHM contents for upstream, midstream and downstream divisions are provided in Table 2. Overall, VHMs made up around 25–30 wt% of the THM content with other heavy minerals such as amphibole, pyroxene, kyanite, sillimanite, monazite, apatite and xenotime making up the remaining 70–75 wt%. Based on VHM content data (not shown) determined at individual sample locations, a distribution pattern showing the variation in VHM concentration was generated by plotting the coordinates of the sample locations on a GIS map and then overlaying the VHM concentration percentages (Figure 4). Although the THM distribution (Figure 3c) revealed no preferential concentration of heavy minerals, the VHM distribution map shows that the upstream division of the studied area appears to be the most prospective for valuable heavy minerals.

Table 2. The average THM concentration and abundance of VHM within the three stream divisions.

Sample ID	THM (wt%)	Magnetite (wt%)	Ilmenite (wt%)	Zircon (wt%)	Garnet (wt%)	Heavy Others (wt%)
Upstream	10.42	2.6	5.41	2.02	8.04	81.93
Midstream	11.02	2.8	4.56	2.07	8.90	81.67
Downstream	10.75	2.3	4.29	2.32	7.73	83.36
Average	10.73	2.57	4.75	2.14	8.22	82.32

**Figure 4.** Map showing the concentration of VHM from each sample location. In general, VHM contents were highest in the upstream region.

3.2. Beneficiation Studies for Recovery of VHM

Bench scale separation techniques based on exploiting the physical properties of the minerals were used to provide sufficient material for characterisation studies of the more valuable ilmenite- and zircon-rich fractions. These included standard gravity, magnetic and electrostatic separations, as practised in the heavy mineral sands industry.

The sand samples from each location were processed by wet gravity separation using a Wilfley shaking table for the separation of heavy and light fractions and recovery of VHMs. The average amount of VHMs recovered via the shaking table were considerably less than obtained via heavy liquid separation. This was because the heavy liquid procedure recovered all minerals with a SG > 2.89 and therefore included additional non-valuable heavy minerals such as amphibole, pyroxene, kyanite and sillimanite. In comparison, the use of the shaking table allowed a better split between heavy and light minerals ensuring that the concentrate was dominated by the VHM and there was greater rejection of gangue minerals.

The individual concentrates recovered from the gravity separation tests on each sample were insufficient to run further individual recovery tests. For this reason, the gravity concentrates from all samples of each stream section (up-, mid- and downstream) were combined and thoroughly mixed to make three composite samples. These concentrates were each subjected to IRMS and then ESPS dry separation procedures for the recovery of six separate VHM fractions according to the flowsheet in Figure 2. A summary of the separation results is given in Table 3 while results for the average mass recovery to each of the individual fractions is given in Table 4. Results indicate that garnet and ilmenite make up the bulk of the HM in the Brahmaputra River sand at concentrations of 0.88 and 0.51 wt%, respectively, with recoveries of 83.6% for garnet and 92.6% for ilmenite. Magnetite (77.6% recovery) and zircon (97.4%) were also recoverable but were present at much lower levels of 0.28 wt% and 0.23 wt%, respectively. The lower magnetite recovery was attributed to its finer particle size and presence as composite particles [18]. The physical properties of garnet and other ferrosilicate minerals, such as amphibole and pyroxene group minerals, are similar and the garnet tended to separate with these silicate minerals.

Table 3. Results of IRMS and ESPS separations conducted on the combined VHM concentrates from the Wilfley shaking table.

Streamline ID	VHM Concentrate (wt%)	Composition of the Concentrate					
		Magnetite (wt%)	Ilmenite (wt%)	Zircon (wt%)	Garnet (wt%)	Magnetic Others (wt%)	Non-Mag. Others (wt%)
Upstream	2.75	8.87	20.16	8.32	31.80	25.06	5.79
Midstream	1.47	12.10	27.96	11.80	38.04	8.01	2.09
Downstream	1.76	11.21	22.96	13.48	41.20	8.97	2.18
Average	1.99	10.73	23.69	11.20	37.01	14.01	3.35

Table 4. Summary of results for the average recovery of VHM into the combined concentrate.

Mineral	VHM Concentrate (wt%)	Recovery (%)	Grade (wt%)	Calc. Head (wt%)
Magnetite	0.21	77.6	95.80	0.28
Ilmenite	0.47	92.6	91.14	0.51
Zircon	0.22	97.4	98.05	0.23
Garnet	0.74	83.6	95.45	0.88

A larger scale test program was conducted on a bulk sand sample of 593 kg. The sample was a combined sample made of ~9–10 kg of sand from each sample site. The sand was processed according to the flowsheet in Figure 2 and results, including assay results and mass recoveries, are shown in Figure 5. Weights for each of the recovered fractions were as follows: Magnetite—1.0 kg (0.17 wt%); Ilmenite—0.5 kg (0.08 wt%); Garnet—1.1 kg (0.19 wt%); Zircon—0.2 kg (0.03 wt%); Magnetic others—7.7 kg (0.76 wt%); and Non-magnetic others—1.4 kg (0.24 wt%). This is a total mass recovery of VHMs of

1.47 wt%. Further work to optimise the operating conditions of the separation equipment can be expected to increase the mass recovery of VHMs.

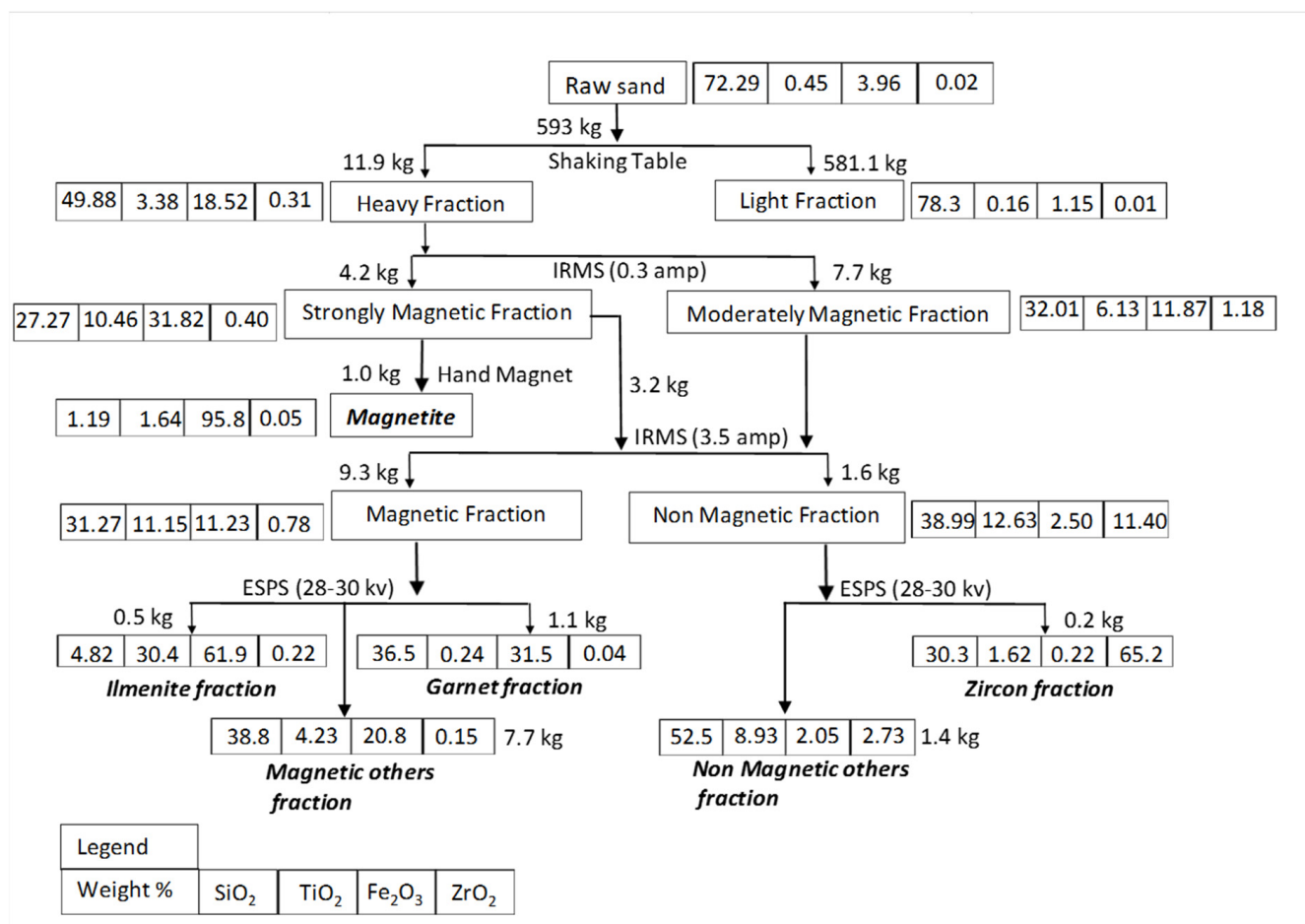


Figure 5. Process flow sheet with material balance showing the recovery of VHM from the combined Brahmaputra River sand, Kurigram District, Bangladesh.

3.3. Characterization of VHM Fractions Ilmenite, Zircon, Garnet and Magnetite

3.3.1. Chemical Analysis by XRF

The XRF data for the recovered VHM bulk samples representing the magnetite, ilmenite, garnet, zircon, magnetic others and non-magnetic other fractions, are listed in Table 5.

The XRF results for the ilmenite fraction indicated the concentrate contained 30.4 wt% TiO₂ and 61.9 wt% Fe₂O₃. Other elements present included: silicon (4.82 wt% SiO₂), aluminium (1.88 wt% Al₂O₃), calcium (1.00 wt% CaO), magnesium (0.59 wt% MgO) and manganese (1.10 wt% Mn₃O₄). The low TiO₂ content as well as the presence of these oxides/elements as additional components in the concentrate suggest silicate and aluminosilicate gangue phases were present while the high Fe₂O₃ (61.9 wt%) suggests a high magnetite/hematite or titano-magnetite/hematite component, either in solid solution or as separate contaminant grains.

Table 5. Bulk chemical analysis results (XRF) determined for the ilmenite, zircon, magnetite, garnet, magnetic others and non-magnetic other fractions.

Component (wt%)	DL *	Ilmenite	Zircon	Magnetite	Garnet	Mag. Others	Non-Mag. Others
TiO ₂	0.005	30.4	1.62	1.61	0.24	4.23	8.93
Fe ₂ O ₃	0.005	61.9	0.22	99.7	31.5	19.82	2.05
SiO ₂	0.01	4.82	30.3	1.31	36.5	38.9	52.5
Al ₂ O ₃	0.01	1.88	0.52	0.68	20.8	16.4	8.26
CaO	0.005	1.00	1.94	0.30	5.56	11.1	14.2
Cr ₂ O ₃	0.005	0.180	<DL	0.300	0.023	0.060	0.030
K ₂ O	0.005	0.10	0.034	0.037	0.066	0.490	0.480
MgO	0.01	0.59	<DL	0.21	3.64	4.90	0.78
Mn ₃ O ₄	0.005	1.10	0.03	0.29	2.37	0.77	0.06
Na ₂ O	0.01	0.10	0.33	0.01	0.16	0.84	1.01
P ₂ O ₅	0.005	0.09	1.01	0.067	0.072	0.28	5.47
ZrO ₂	0.005	0.22	65.2	0.038	0.036	0.15	2.73
Th (ppm)	20	20	440	<20	140	150	140
U (ppm)	20	<20	700	<20	<20	<20	40

* DL = detection limit for each component. <DL = below detection limit.

The zircon fraction contained 65.2 wt% ZrO₂ and 30.3 wt% SiO₂ which was close to stoichiometric zircon which contains of 67.2 wt% ZrO₂ and 32.8% SiO₂. Major impurities in the concentrate included titanium (1.62 wt% TiO₂), which was likely present as the mineral rutile and calcium (1.94 wt% CaO) and phosphorus (1.01 wt% P₂O₅), oxides commonly associated with apatite. Minor impurities included aluminium (0.52 wt% Al₂O₃), sodium (0.33 wt% Na₂O) and iron (0.22 wt% Fe₂O₃). Aluminium and sodium may be associated with clays and/or weathered feldspars while iron may be associated with rutile and/or ilmenite. Note that, compared to all other VHM fractions, zircon contains elevated levels of uranium and thorium at values of 700 ppm U and 440 ppm Th. In terms of producing a commercial zircon product, these levels are problematic as combined U+Th levels in marketed zircon concentrates typically need to be below 500 ppm [25].

The magnetite fraction was characterised by high Fe₂O₃ (99.7 wt%) with minor impurities being TiO₂ (1.61 wt%) most likely as ilmenite or titanomagnetite components and SiO₂ (1.31 wt%). Examination of magnetite grains in Brahmaputra sediments by Rahman et al. [18] showed that magnetite often formed complex intergrowths with silicate and aluminosilicate gangue minerals and the presence of these grains is likely responsible for the high SiO₂ values and the contamination by aluminium (0.68 wt% Al₂O₃), calcium (0.30 wt% CaO), magnesium (0.21 wt% MgO) and manganese (0.29 wt% Mn₃O₄). Minor contamination by chromium (0.30 wt% Cr₂O₃) and vanadium (0.28 wt% V₂O₅) is primarily due to solid solution of these oxides into magnetite (some Al₂O₃ may also be present as a solid solution component).

The garnet fraction contained, on average 36.5 wt% SiO₂ and 20.8 wt% Al₂O₃ which is within the range of alumina and silica values typically observed for garnets within the four end members of the almandine(Fe)-pyrope(Mg)-grossular(Ca)-spessartine(Mn) isomorphous series. A garnet corresponding in composition with any one endmember is rare, however and the name is usually ascribed according to the dominant 'molecular' type present. Average Fe₂O₃ (31.5 wt%), CaO (5.56 wt%), MgO (3.64 wt%) and Mn₃O₄ (2.37 wt%) assays indicate that the garnets present in the fraction are most likely almandine (i.e., Fe-rich) with minor, in order of decreasing abundance, grossular, pyrope and spessartine

components. The levels of CaO and Fe₂O₃ may also include contributions from calcium and iron aluminosilicate heavy minerals such as amphibole and epidote.

The magnetic others fraction was, as expected, iron-rich (19.82 wt% Fe₂O₃) with considerable amounts of SiO₂ (38.9 wt%), Al₂O₃ (16.4 wt%), CaO (11.1 wt%) and MgO (4.90 wt%). The analyses are consistent with the bulk of the magnetic others fraction comprising heavy mineral aluminosilicates such as amphiboles, garnets, epidotes that are typically present in Brahmaputra River sands [18]. The minor amount of TiO₂ (4.23 wt%) indicates possible incorporation of ilmenite in the fraction that could potentially be recovered with further refinement of the process flowsheet. Both Na₂O and K₂O were low (<1 wt%) indicating low amounts of feldspar in the fraction. Quantitative XRD analysis of the fraction indicated the sample mineralogy was dominated by amphibole (31 wt%), epidote (25 wt%), garnet (24 wt%) and quartz (8 wt%). Minor phases included ilmenite (4 wt%), titanite (5 wt%), mica (1 wt%) and trace amounts of chlorite and clay. In comparison, the non-magnetic others fraction was high in SiO₂ (52.5 wt%), CaO (14.2 wt%) and Al₂O₃ (8.26 wt%) implying the presence of quartz, feldspars and aluminosilicates, while high TiO₂ (8.93 wt%) suggests possible rutile incorporation. High P₂O₅ (5.47 wt%) suggests the presence of apatite and/or monazite and 2.73 wt% ZrO₂ indicates likely zircon grains. The QXRD analysis of the fraction indicated 55 wt% quartz, 15 wt% plagioclase, 10 wt% fluorapatite, 7 wt% titanite, 5 wt% epidote and 4 wt% potassium-rich feldspar. Minor to trace amphibole (2 wt%), mica (2 wt%) and chlorite (<1 wt%) were also present.

In the following sections, a more detailed examination of the four fractions ilmenite, zircon, garnet and magnetite, which contain the VHM, is provided with discussion aimed at improving their overall recovery.

3.3.2. Ilmenite Fraction

The results of the phase identification and QPA (relative crystalline wt%) indicated a high proportion of the Fe-rich phases, ilmenite (44.9 wt%) and hematite (42.5 wt%), consistent with the high Fe₂O₃ and the low TiO₂ assays reported in Table 5. Both minerals are susceptible to recovery to the magnetic fraction; however, it is unclear from the QXRD results as to whether these phases are present as discrete phases or composite particles. Minor rutile (2.1 wt%) and zircon (0.3 wt%), other potentially recoverable valuable minerals, were also present. It is possible these represent mineral entrainment or minerals occurring as composite phases or inclusions. Given rutile and zircon inclusions are typically associated with ilmenite, we suggest it is most likely the latter. Amphibole (4.1 wt%), mica (2.5 wt%), feldspar (1.7 wt%), quartz (1.1 wt%) and kaolinite (0.7 wt%) were the main gangue mineral phases. These contribute significantly to the elevated SiO₂ and Al₂O₃ levels reported for the bulk assay. Based on previous work by Rahman et al. [26] there is also likely to be minor garnet present (although if present, it is below the detection limit of QXRD). The gangue mineral suite is also consistent with the observed assays for the minor elements CaO (amphibole, plagioclase feldspar, garnet), K₂O (mica, feldspar), MgO (amphibole, garnet) and Na₂O (plagioclase feldspar).

Examination of the ilmenite fraction by BSE imaging confirmed the Fe-Ti oxide and gangue mineralogy indicated by QXRD. Typical textures of the Fe-Ti oxide phases are shown in Figure 6a,b. In these images, homogeneous hematite (Figure 6b), titanohematite (Ti-Hem_{ss}) and ilmenite grains were all observed; however, a universal feature was the abundance of grains exhibiting complex exsolution of ilmenite in Ti-Hem_{ss} hosts and of hematite or Ti-Hem_{ss} exsolution in ilmenite hosts. These grains were characterised by thick bands of either ilmenite (dark bands -Figure 6c) or Ti-Hem_{ss} (light bands) which contain tiny droplets, lenses or rod-like bodies and lamellae of hematite in the ilmenite matrix, or ilmenite in the Ti-Hem_{ss} matrix (see Figure 6a,b). As discussed recently by Rahman et al. [26] at temperatures above 800 °C there is complete miscibility (solid solution) between ilmenite and hematite; however, during crystallisation and cooling, hematite and ilmenite become segregated forming titanohematite, which is characterised by lamellae of hematite in varying amounts intercalated with ilmenite.

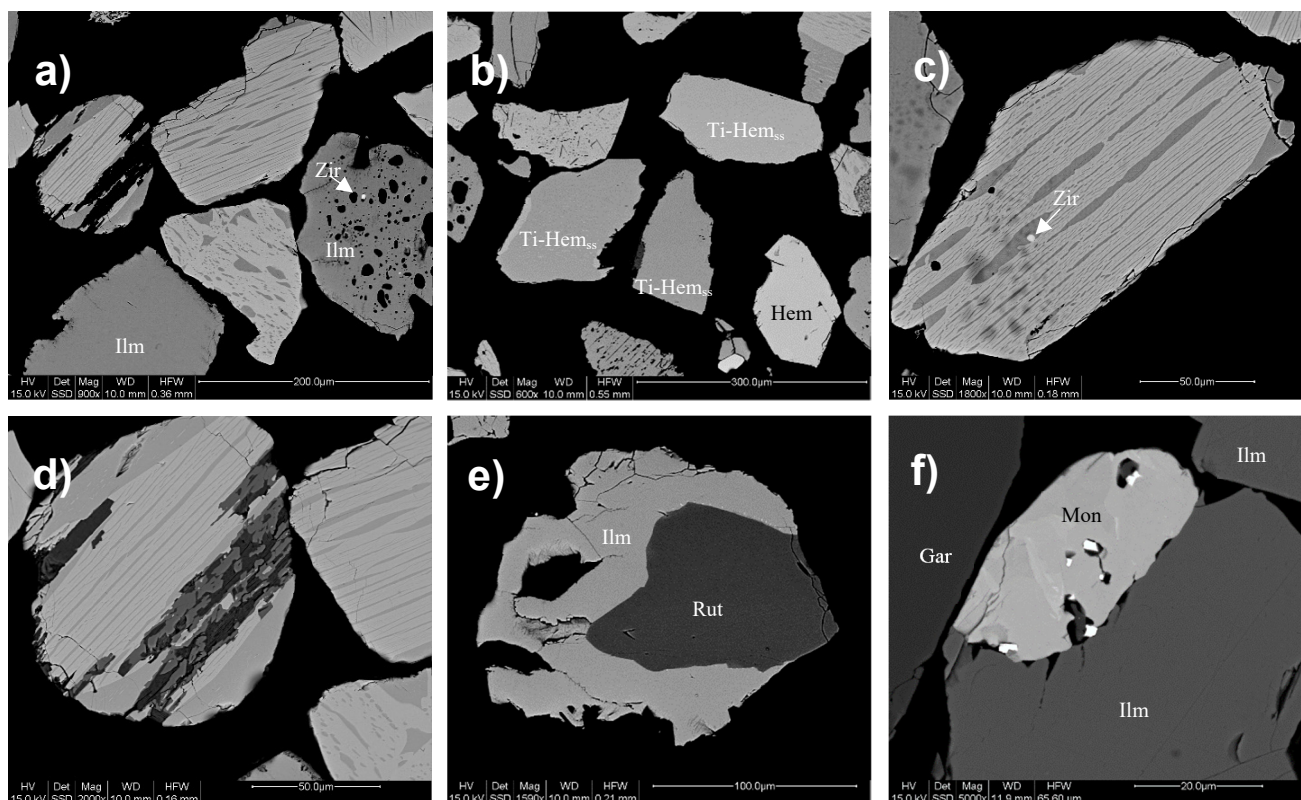


Figure 6. Back-scattered electron (BSE) images of the bulk ilmenite sample showing textures of the various Fe-Ti oxide particles as well as images showing grains with inclusions of potentially valuable heavy minerals. (a–c) show typical ilmenite textures with grains showing examples of titanohematite (Ti-Hem_{ss}) exsolution (banded grains), homogeneous ilmenite grains (also in (e,f)) as well as homogeneous hematite grains (c). The presence of primary, unaltered inclusions (e.g. quartz in the highly porous-looking ilmenite towards the top left of (a)) and sphene (titanite) and garnet incorporation in the exsolved ilmenite shown in (d). Inclusions of other valuable minerals typically associated with heavy mineral Fe-Ti oxides such as rutile (e), zircon (small, bright spots in (a,c)) and monazite (f) were also noted. Key to abbreviations used: Ilm = ilmenite; Ti-Hem_{ss}—solid solution between titanohematite and hematite; Hem = hematite, Rut = rutile, Gar = garnet, Zir = zircon and Mon = monazite. Note that the small very high contrast grains associated with monazite in (f) represent ThO₂ grains.

Many of the Fe-Ti oxide grains were angular to sub-angular in appearance consistent with proximity to source. Further evidence for little weathering/degradation includes the presence of primary, unaltered inclusions (e.g., quartz) and sphene (titanite) and garnet (Figure 6d). Inclusions of other valuable minerals typically associated with heavy mineral Fe-Ti oxides such as rutile (Figure 6e) zircon (small, bright spots in (Figure 6a,c)) and monazite (Figure 6f) were also noted.

Results from EPMA mapping are provided in Figure 7. They confirm the main impurities in the ilmenite fraction included amphibole, mica, feldspar and quartz. These were typically present as discrete grains indicating that further separation may be possible to generate a purer ilmenite concentrate. The results also show that the current beneficiation procedure leads to a mixed Fe- and Fe-Ti oxide concentrate that has low TiO₂ and high Fe₂O₃ (reflecting the high proportion of hematite and complex Ti-Hem_{ss} particles). There is a need, therefore, to refine the beneficiation procedure, if possible, to separate hematite from ilmenite and from Ti-Hem_{ss} (which could be used in a different stream that would recover both Ti and Fe, separately). The suggested separation testing procedure would likely be based on low intensity magnetic separation (LIMS) using equipment such as a Davis tube apparatus. If complete separation was not achievable, it is likely that the Fe-Ti concentrate would need to be processed in a high temperature smelting (slag) process that would generate pig iron and TiO₂-enriched slag products [27].

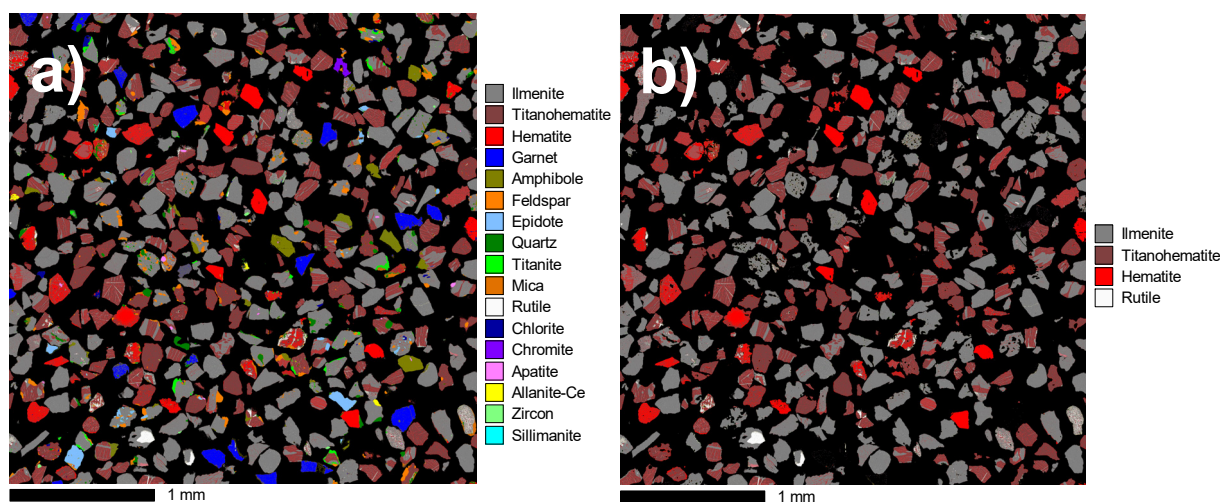


Figure 7. EPMA map results for the bulk ilmenite sample showing (a) the phase patched map with the distribution of all phases present in the sample and (b) a map with the distribution of Fe- and Fe-Ti oxide phases and rutile. Operating conditions for the microprobe during mapping were an accelerating voltage of 20 keV, a beam current of 80 nA, a step size of 2 μm (in x and y) and a counting time of 25 ms per step.

3.3.3. Zircon Fraction

The QXRD results for the zircon fraction indicated the sample comprised 95.6 wt% zircon, 2.0 wt% apatite, 1.3 wt% quartz and 1.1 wt% rutile. The latter three phases contribute to the elevated levels of CaO, P_2O_5 , TiO_2 and SiO_2 observed in the XRF results in Table 5. Results from EPMA mapping are provided in Figure 8. The map results confirm the sample comprised >95% (by area) of zircon particles with apatite and sillimanite being the main impurity phases along with minor titanomagnetite, rutile and quartz. Trace phases present (not all shown) included feldspar, mica, ilmenite and titanite. Many of the extraneous impurity phases are present as discrete grains and so could potentially be removed through further processing. For example, in some operations, zircon concentrate can be passed through an additional gravity separation circuit (spirals) to remove the lower SG material (quartz, sillimanite, apatite) from the higher SG zircon. Further electrostatic separation after gravity separation removes residual conductors from the zircon and traces of monazite/xenotime (if present) are removed with induced roll magnets. A final pass across an air table can remove fine quartz and residual sillimanite that has not been rejected by the gravity separation circuit. Note, however, there are also a considerable number of zircon grains that contain small inclusions of phases such as apatite (dominant inclusion phase), quartz, mica and ilmenite. These would not be removed through additional dry processing procedures.

3.3.4. Garnet Fraction

QXRD analysis results for the garnet fraction indicated the sample contained 79 wt% garnet, 9 wt% amphibole, 8 wt% epidote, 3 wt% quartz and 1 wt% mica. EPMA mapping results are shown in Figure 9. The data indicate the sample comprised over 81.1% (by area) garnet and with minor amounts of, in order of relative abundance, amphibole (4.6%), epidote (4.4%), Ce-allanite (3.6%) and quartz (1.8%). Element distribution plots (not shown) indicated the garnets varied considerably in composition although in general, most were Fe-rich. Compared to XRD, the EPMA analysis was capable of identifying a significant number of minor mineral phases including biotite, ilmenite, titanate, magnetite, apatite, feldspar, sillimanite and zircon, plus trace sulphides. Only the presence of phases with abundance >1% are shown in Figure 9. For the most part, the garnet grains exhibited a high degree of angularity and were largely inclusion free. Where inclusions were observed, these tended to be dominated by phases such as quartz, amphibole and epidote.

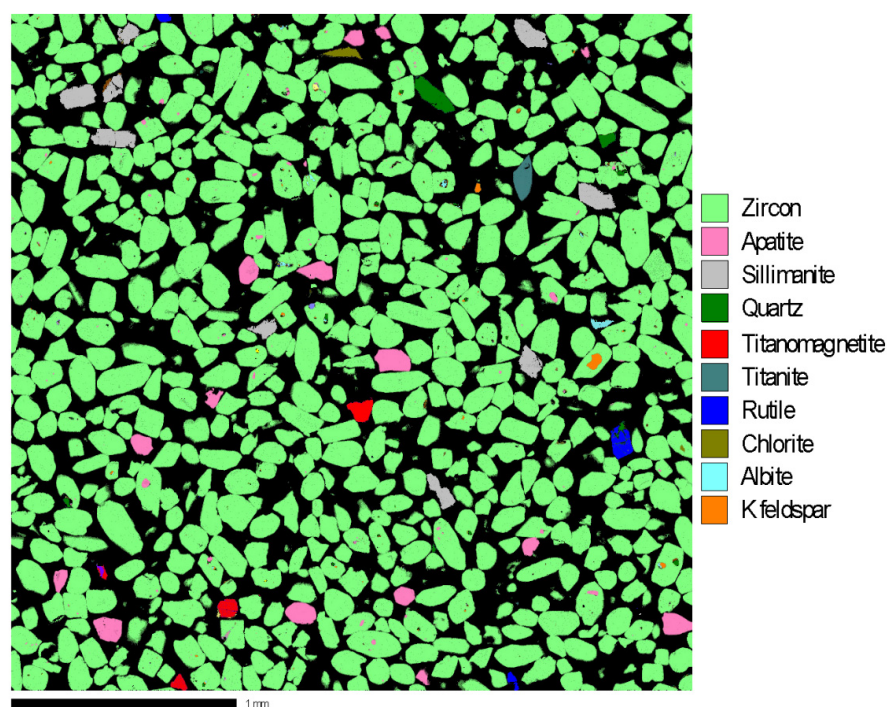


Figure 8. EPMA map results for the zircon concentrate showing the phase patched map with the distribution of all phases present in the sample. Operating conditions for the microprobe during mapping were an accelerating voltage of 15 keV, a beam current of 80 nA, a step size of 3 μm (in x and y) and a counting time of 30 ms per step.

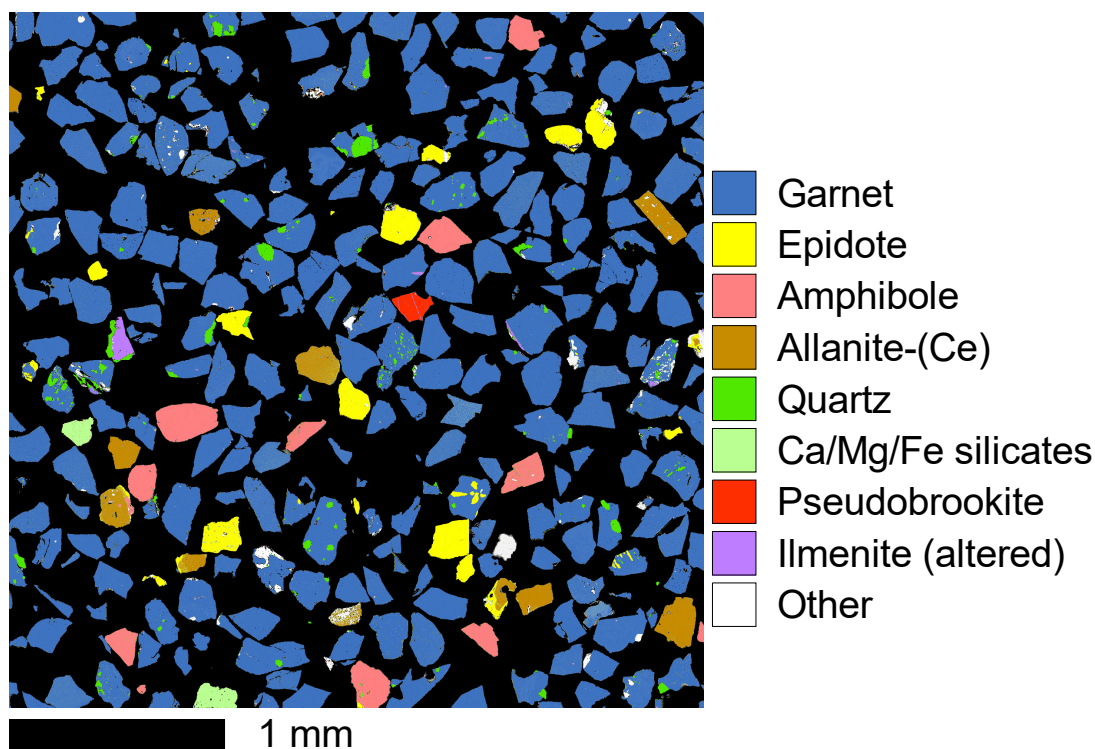


Figure 9. EPMA map results for the garnet concentrate showing the phase patched map with the distribution of all phases present in the sample. Only phases with an abundance of >1% by area are shown. Trace phases are labelled as ‘Other’. Operating conditions for the microprobe during mapping were an accelerating voltage of 20 keV, a beam current of 80 nA, a step size of 2 μm (in x and y) and a counting time of 15 ms per step.

Approximately 126 individual garnet grains were analysed by quantitative WD-based EPMA. The Brahmaputra River garnets contained, on average, SiO₂ (34.1–38.7 wt%), FeO (10.9–38.8 wt%), Al₂O₃ (20.5–24.3 wt%), CaO (0.4–23.4 wt%), MgO (0.1–7.9 wt%) and MnO (0.1–15.7 wt%). The compositional data were re-cast into the end-member molar components almandine (Alm), pyrope (Pyr), spessartine (Spess) and grossular (Gro) and the data plotted on a ternary Alm-Pyr-(Spess+Gro) plot in Figure 10. In addition to, a few (Spess+Gro)-rich garnet outliers, most of the data plot toward the Almandine-rich apex. Based on all measured garnet compositions the average composition was Alm₆₈Pyr₁₃Gro₁₃Spe₆.

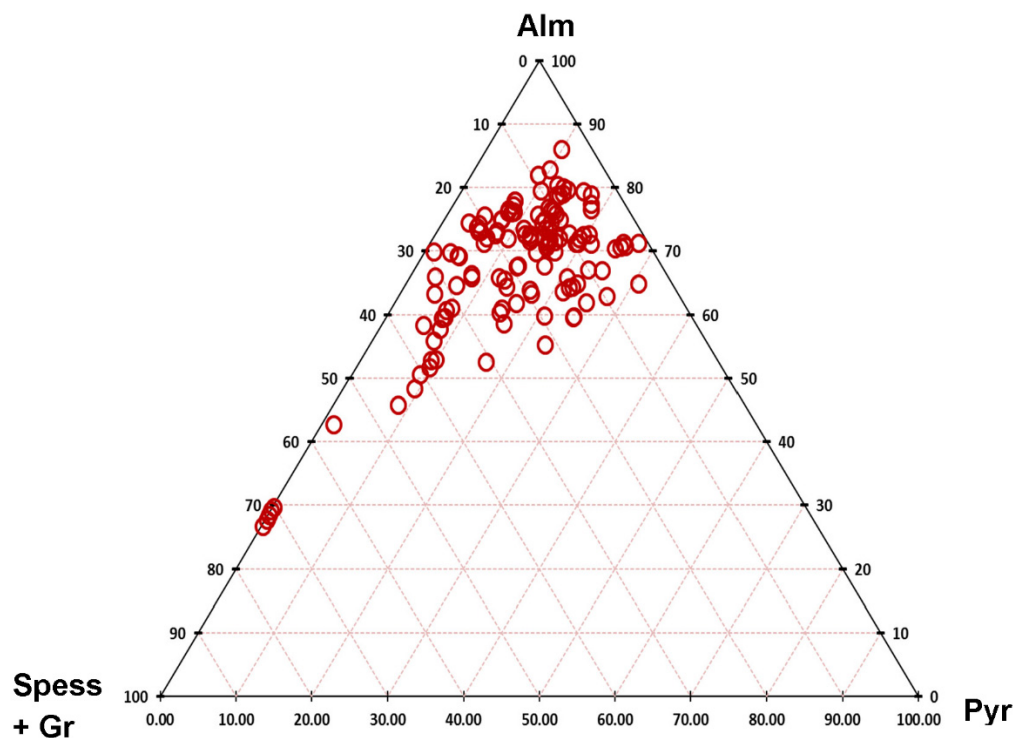


Figure 10. Ternary Alm-Pyr-(Spess+Gr) plot showing the range in garnet compositions from the northern Brahmaputra River sands.

Almandine-rich garnet is the principal abrasive for industrial applications [28] because of its high melting point (~1320 °C), high specific gravity (4.3) and high hardness (7–7.5 Mohs scale). Spessartine- and pyrope-rich garnets have also been used as industrial-grade garnet abrasives such as air-blasting [29]. Garnet is the main substitute for quartz sand which is becoming less used for air-blasting because of the silicosis risk. The average compositional data for the garnets from the northern Brahmaputra River sands are almandine-rich suggesting good potential for air-blasting applications. Most commercial garnet abrasives are within the size range 180–250 micron [30] which is towards the upper limit of the Brahmaputra River garnets based on data in Rahman et al. [18] and additional characterisation test work will be required to determine the grainsize distribution in a fully separated garnet concentrate. As well, further beneficiation will likely be required to remove the additional quartz and aluminosilicate gangue phases (amphibole and epidote) to ensure the concentrate meets commercial specifications.

3.3.5. Magnetite Fraction

The QXRD analysis results for the magnetite fraction showed the sample consisted of 93 wt% magnetite, 3 wt% ilmenite, 1 wt% amphibole, 1 wt% quartz and minor mica, hematite, epidote, feldspar and zircon. EPMA map results (Figure 11) confirm the high content of magnetite (86.1% by area) along with minor phases such as Fe oxide (3.6%, most

likely as hematite), ilmenite (3.0%), amphibole (1.6%), a Cr-rich Fe oxide (1.3%, possibly chromite) and epidote (0.8%). Phases lower in abundance than ~0.8% included quartz, garnet, biotite, feldspar, apatite, titanite, zircon and sulphides.

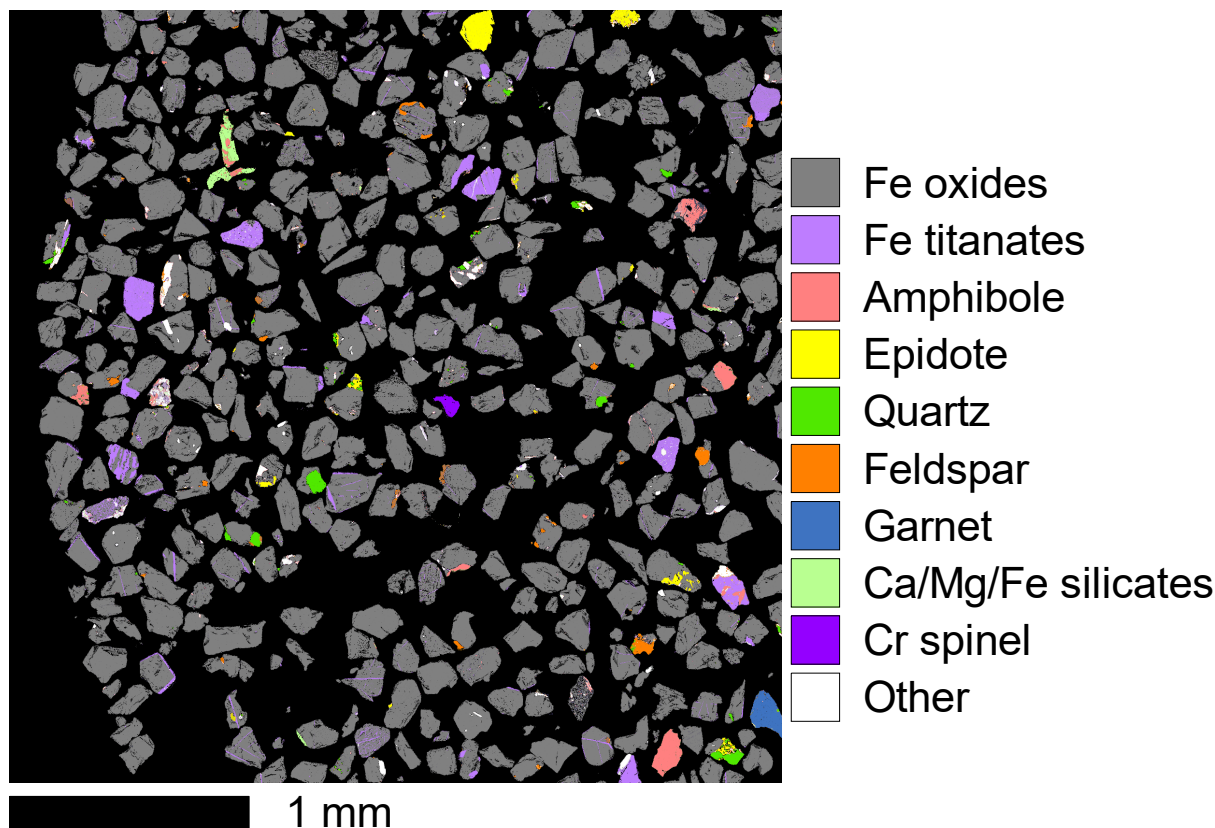


Figure 11. EPMA map results for the magnetite concentrate showing the phase patched map with the distribution of all phases present in the sample. Only phases with an abundance of >1% by area are shown. Trace phases are labelled as ‘Other’. Operating conditions for the microprobe during mapping were an accelerating voltage of 20 keV, a beam current of 80 nA, a step size of 2 μm (in x and y) and a counting time of 15 ms per step.

The magnetite particles in the river sands occurred as a mixture of fully liberated particles to particles that formed complex intergrowths with silicate gangue minerals. Most of the magnetite particles were angular in appearance indicating a resistance to abrasion during transport. Individual magnetite particles exhibited textures varying from optically homogeneous, Ti-poor and without discernible zoning or exsolution, to particles dominated by exsolution of ilmenite lamellae (elongated parallel to 0001 planes). Rare titanomagnetite particles were also present. Similar textures among magnetite grains in Brahmaputra River samples were noted previously by Rahman et al. [18]. Separation of the magnetic-rich particles versus the more complex particles with exsolved ilmenite lamellae in order to generate a pure magnetite concentrate for commercial application may be difficult; however, LIMS testing is recommended as an initial starting point. Similar to the complexities of the mineral assemblage observed in the ilmenite fraction, it is recommended that further work be undertaken to determine the optimum conditions for separation of all components.

4. Conclusions

Analysis of samples collected from sand bars within the northern Brahmaputra River sands indicated the average THM content across all sample sites was 10.73 wt%. The THM concentrations were highest at the outermost edges of the river and lowest within the mid-channel regions while there was no variation in THM content from north (upstream)

to south (downstream). The VHM made up around 25–30 wt% of the THM content and a VHM distribution map showed that the upstream areas appeared to be the most prospective for VHM.

Bench scale separation techniques were used to provide sufficient material for characterisation studies of the more valuable ilmenite-, zircon- and garnet-rich fractions. The ilmenite fraction was composed of three main grain types: homogeneous ilmenite; grains exhibiting complex exsolution of ilmenite in Ti-Hem_{ss} hosts and of hematite or Ti-Hem_{ss} exsolution in ilmenite hosts and hematite. There is a need to refine the beneficiation procedure to separate these three grain types. The zircon fraction comprised 95.6% zircon with minor apatite, sillimanite, quartz and rutile. The impurity phases were largely present as discrete grains so could potentially be removed through further processing. Mineralogical analysis of the garnet fraction indicated the sample contained 79% garnet, with 9% amphibole, 8% epidote, 3% quartz and 1% mica. Compositions determined on >100 individual garnet grains indicated the garnets were mostly almandine-rich with ~68% almandine component. Further beneficiation will be required to remove the additional quartz and aluminosilicate gangue phases (amphibole and epidote) to ensure the concentrate meets commercial specifications.

This study has demonstrated that it is possible to recover VHM components from the Brahmaputra River sands but more in-depth studies with samples from different regions in the basin are necessary to identify prospective areas and further work is necessary to develop more efficient beneficiation processes for the VHMs.

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