

# **Characterization and Recovery of Heavy Minerals from Luís Correia Distric, Piauí**

## **Caracterização e Aproveitamento dos Minerais Pesados da Região de Luís Correia, Piauí**

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### **Abstract**

Two samples of heavy minerals from beach sand were collected in Piauí. According to the dense liquid separation results, the total heavy minerals content of the samples ranged from 6.45% to 10.14%. The minerals of interest (ilmenite, rutile, zircon and monazite) are mostly concentrated in the -74+53  $\mu\text{m}$  size fraction. Beneficiation tests using screening followed by gravity separation, two stages of magnetic separation and one step of electrostatic separation demonstrated the non-individual separation of the heavy minerals specimens, being the Products 2 and 3 the fractions with the higher heavy mineral content and 64.5% and 59.6% metallurgical recovery, respectively. The quartz contamination after the electrostatic separation confirmed the necessity of a complementary stage of beneficiation by gravity separation.

Key-words: Characterization, Heavy Minerals, Recovery.

### **Resumo**

Duas amostras de minerais pesados de areia de praia foram coletadas no Piauí. De acordo com os resultados da separação em líquido denso, o teor de minerais pesados varia de 6,45 a 10,14%. Os minerais de interesse (ilmenita, rutilo, zirconita e monazita) são concentrados prioritariamente na fração granulométrica -74+53  $\mu\text{m}$ . O circuito de beneficiamento composto por peneiramento, separação gravítica, separação magnética em duplo estágio e, separação eletrostática possibilitou inferir que não houve separação individual dos minerais pesados, sendo os Produtos 2 e 3 as frações com teor mais elevado de minerais pesados, 64,5% e 59,6% de recuperação metalúrgica, respectivamente. A contaminação por quartzo depois da separação eletrostática confirmou a necessidade de adição de uma posterior etapa de separação gravítica.

Palavras-chave: Caracterização, Minerais Pesados, Aproveitamento.

### **1. Introduction**

The main characteristic of heavy minerals is that their specific gravity (SG) is higher than that of common and/or gangue minerals. They can originate from igneous, metamorphic, and sedimentary rocks and are concentrated in

coastal areas. In Brazil, heavy minerals deposits are still barely explored, being restricted to the Guaju mine (Cristal Group) located in the state of Paraíba. The main minerals beneficiated are ilmenite ( $\text{FeTiO}_3$ ) and zircon ( $\text{ZrSiO}_4$ ). Previously, Brazilian Nuclear Industries (INB) produced ilmenite, zircon, rutile ( $\text{TiO}_2$ ), and monazite  $[(\text{Ce},\text{La},\text{Nd},\text{Th})\text{PO}_4]$  concentrates aiming to their *in natura* commercialization (SCHNELLRATH et al., 2001) regarding the importance of beach sands as a source of rutile, ilmenite, and zircon, and their worldwide uses. Usually, heavy minerals from beach sand are concentrated firstly using gravity separation (SAMPAIO et al., 2001; SCHNELLRATH et al., 2001; LAXMI et al., 2013), magnetic separation, electrostatic separation, and also flotation (ROUTRAY et al., 2013).

## 2. Objective

Characterize the heavy minerals from the municipality of Luís Correia, aiming a technological development for their economical uses, and suggest a preliminary beneficiation process to separate and concentrate, individually, the minerals specimens such as ilmenite, rutile, zircon and monazite.

## 3. Materials and Methods

Samples, from two different localities of Luís Correia district, Piauí, were firstly prepared and analyzed by X-Ray fluorescence spectrometry (Panalytical Axios Max Rh emission) and X-Ray diffraction (D4 Endeavor Co emission). Then, the samples were after wet-screened using 425, 300, 212, 150, 106, 74, 53 and 44  $\mu\text{m}$  apertures and each fraction also analyzed by XRF and XRD. Other physical characteristics were measured such as bulk density and specific gravity (Pycnometer method). After, the samples were subject to the dense liquid separation test with bromoform (SG 2.89) followed by the methylene iodide (SG 3.32). One sample was chosen considering its higher Total Heavy Minerals (THM). The methylene iodide sink fraction ( $-150 \mu\text{m}$ ) of the chosen sample was separated using, firstly, a hand magnet and, posteriorly, a Frantz Separator (model L-1 Isodynamics, forward slopes  $15^\circ$ ) with magnetic field strengths of 0.150, 0.450, 0.675, 0.950 and 1.300 T. Each fraction was also analyzed in detail using a scanning electron microscope (Hitachi TM 3030 Plus Tabletop Microscope), XRF and XRD.

The preliminary beneficiation flowsheet was composed of a screening stage (cut-off size  $150 \mu\text{m}$ ), gravity separation using shaking table (model Super Duty Diagonal Deck), magnetic separation 1<sup>st</sup> stage at 0.150 and 0.650 T magnetic field strengths (Boxmag Rapid), magnetic separation 2<sup>nd</sup> stage at 1.000 T magnetic field strength (Inbras Magnetic Roll Separator model RE-05/4-1) and electrostatic separation (Inbras Separator model ES-14/01S); products generated in each beneficiation step were quantified by Rietveld method and XRF.

## 4. Results and Discussions

The physical properties of the samples and the preliminary results for ore concentration are given in Table 1. An average of 64%, by weight, of the sink fraction from methylene iodide is composed of minerals with low magnetic susceptibility (up to 1.300 T) as ilmenite, epidote, monazite, actinolite and staurolite. The nonmagnetic fraction

contains zircon, rutile, kyanite, anatase, sillimanite and quartz. The greater part of the samples is recovered in the range of 0.450 to 0.675 T magnetic field strength.

Table 1. Physical properties of samples.

Property	Sample 1	Sample 2
Bulk density and SG of raw material (g/cm <sup>3</sup> )	1.46 and 2.73	1.62 and 2.76
d <sub>80</sub> passing size (µm)	190	400
Total heavy minerals - THM (%)	6.45	10.14
Heavy minerals - HM (SG > 3.32) (%)	4.03	8.82
Heavy minerals (2.89 < SG < 3.32) (%)	2.42	1.33
Total high magnetic heavy minerals – THMHM <sup>1</sup> (%)	1.50	1.70
Total magnetic heavy minerals – TMHM <sup>2</sup> (%)	65.1	63.1
Total nonmagnetic heavy minerals – TNHM (%)	33.4	35.2
Specific gravity of sink fraction of methylene iodide (g/cm <sup>3</sup> )	4.17	4.36

Notes: <sup>1</sup> Minerals recovered using the hand magnet and the Frantz Separator with magnetic field strength of 0.150 T.

<sup>2</sup> All minerals which have a SG higher than that of methylene iodide and which are recovered from 0.450 to 1.300 T.

According to Table 1, regarding the samples THM, only the Sample 2 was characterized and tested. Minerals such as monazite, ilmenite, rutile, and zircon are concentrated preferentially in the -150 µm size fraction. Thus, -150+53 µm is the size fraction where the greater part of the minerals of economic interest is concentrated. The coarse fraction (+150 µm) is composed of quartz, kyanite, staurolite, microcline and albite. The elemental chemical analyze with the principal oxides TiO<sub>2</sub>, ZrO<sub>2</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and REE<sub>oxide</sub> (CeO<sub>2</sub>, La<sub>2</sub>O<sub>3</sub>, Nd<sub>2</sub>O<sub>3</sub> and ThO<sub>2</sub>)+P<sub>2</sub>O<sub>5</sub>, and the mineralogical composition of the raw material is given in Figure 1.

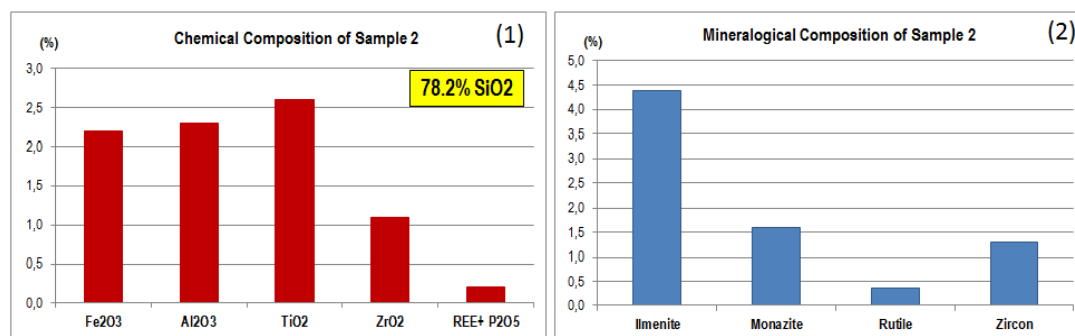


Figure 1. Elemental chemical analyses (1) and the mineralogical composition (2) of sample 2.

As the minerals of interest are in the finer fractions (-150 µm), the magnetic separation of methylene iodide sink fraction by Frantz Separator were carried out on the -150+106 µm, -106+74 µm, and -74 µm size fractions. The -420+150 µm material is considered just in terms of distribution calculus. The -150+106 µm fraction the magnetic fraction comprises ilmenite, staurolite, zircon, and actinolite; the -150+106 µm nonmagnetic fraction is composed mostly of kyanite, zircon, anatase, sillimanite and rutile. The -106+74 µm (0.450 T) fraction contains ilmenite, rutile, and amphibole; the same fraction at 0.675 T is composed of amphibole, ilmenite, epidote, and rutile; and at 0.950–1.300 T of actinolite, staurolite, monazite, ilmenite, and rutile. The nonmagnetic fraction of -106+74 µm is

composed of kyanite, zircon, quartz and rutile. The ilmenite is also the principal mineral of -106+74  $\mu\text{m}$  fraction according to the chemical analysis (57.4%  $\text{TiO}_2$  and 58.1%  $\text{Fe}_2\text{O}_3$  by weight distribution). The -74  $\mu\text{m}$  fraction at 0.450 T is composed of ilmenite and rutile; the same fraction at 0.675 T is composed of actinolite, ilmenite, and rutile; and at 0.950–1.300 T, of monazite, epidote, ilmenite, and rutile; the nonmagnetic fraction of -74  $\mu\text{m}$  is composed of zircon and rutile. The minerals of economic interest as zircon, monazite and rutile are in the -74  $\mu\text{m}$  fraction corresponding to 32.4%  $\text{TiO}_2$ , 68.9%  $\text{ZrO}_2$  and 87.0%  $\text{REE}_{\text{oxide}}+\text{P}_2\text{O}_5$  in terms of material weight distribution.

The ilmenite (Ilm) grains are extremely altered (Figure 2.1), sometimes in pseudorutile (PsRt) and also anatase (Ant) (grain boundaries), as reported by Nair et al. (2009). The existence of ilmenite-pseudorutile grains (Figure 2), due to the reducing conditions of the geological environment (WEIBEL AND FRIIS, 2007), explains the presence of rutile in all XRD pattern of the magnetic products. Zircon grains are fractured and it can influence the quality of the final product, especially with the application of physical processing methods such as screening and scrubbing. The Al-silicates (kyanite and sillimanite) of the nonmagnetic fractions are in the +74  $\mu\text{m}$  size fraction, facilitating their separation by size from the zircon; in other deposits this separation is more complicated (REYNEKE AND VAN DER WESTHUIZEN, 2001) and a flotation stage is needed (ROUTRAY et al., 2013).

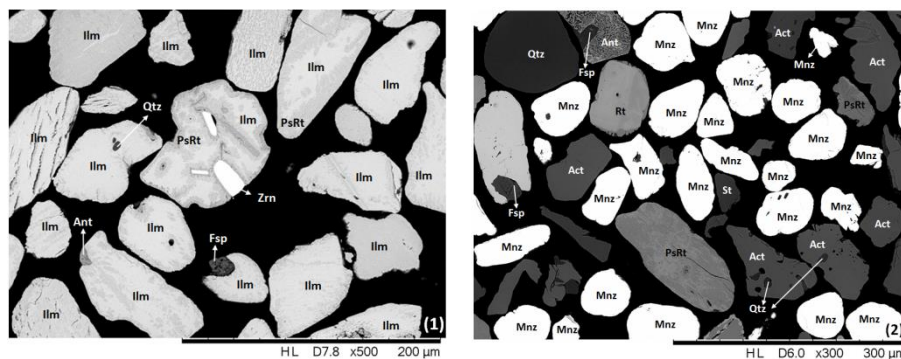


Figure 2. SEM images of magnetic products of sample 2 at 0.450 T (1) and 0.950-1.300 T (2).

The mass balance of Sample 2 processing is presented in Figure 3. The process demonstrated satisfactory results, in terms of metallurgical recovery of HM (ilmenite, monazite, rutile and zircon),  $\text{TiO}_2$ ,  $\text{ZrO}_2$  and  $\text{REE}_{\text{oxide}}+\text{P}_2\text{O}_5$ , for the screening stage and gravity separation, with no losses of Zr- and REE-bearing minerals. However, it was expected a higher ilmenite concentration and recovery in the first step of magnetic separation 1, which was not attained due to ilmenite alteration, being  $\text{TiO}_2$  metallurgical recovery only 13.8% with 59.9% of HM; thus, the **magnetic at 0.150 T** (Product 1) have 25.4% of  $\text{TiO}_2$  and 0.84% of  $\text{P}_2\text{O}_5$ , and do not contain the commercial specifications of 53% of  $\text{TiO}_2$  (minimum) and <0.1% of  $\text{P}_2\text{O}_5$ , as described in SAMPAIO et al. (2001) and Grupo Cristal (2016). It is also possible to see the non-expected concentration of zircon in **magnetic at 0.650 T** fraction (about 60% by weight distribution), probably due to the zircon inclusions in altered ilmenite grains (Figure 2); besides the zircon is a mineral, normally, with a low magnetic susceptibility (Pereira et al., 2005).

The products of magnetic separation 2, **NMag1 magnetic at 1.0T** and **Mag1 magnetic at 1.0T**, contain mostly altered ilmenite (more than 50%), and an average of 2.8% of monazite and 2.9% of rutile by weight (Figure 3).

The **NMag1 magnetic at 1.0T** (Product 2) presented chemical composition of 29.4% TiO<sub>2</sub>, 4.5% ZrO<sub>2</sub> and 3.9% REE<sub>oxide</sub>+P<sub>2</sub>O<sub>5</sub>, and **Mag1 magnetic at 1.0T** (Product 3) of 38.0% TiO<sub>2</sub>, 3.7% ZrO<sub>2</sub> and 4.1% REE<sub>oxide</sub>+P<sub>2</sub>O<sub>5</sub>.

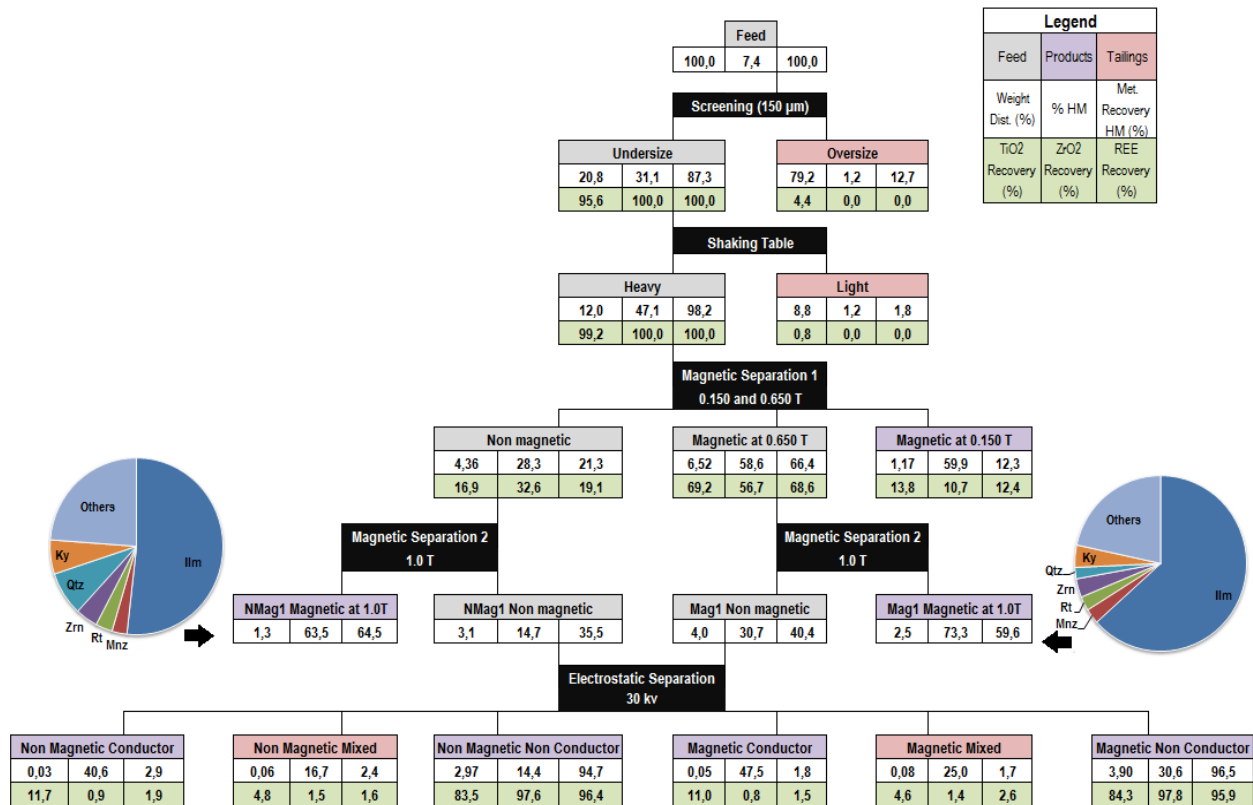


Figure 3. Mass balance of sample 2.

About the products of the electrostatic separation, the **Non Magnetic Mixed** and **Magnetic Mixed** were considered as Tailings. According to the metallurgical recoveries of HM and of previously mentioned oxides during this final stage, it was possible to see the non-effective separation of HM in individual specimens. The **Non Magnetic Conductor** (Product 4) and the **Magnetic Conductor** (Product 5) had unexpressive metallurgical recoveries of 2.9% and 1.8%, respectively; the greater part of both products is composed of rutile (26.5%) and zircon (14%), and the content of monazite is 1.4% and of ilmenite is 2.3%. Their TiO<sub>2</sub>, ZrO<sub>2</sub> and REE<sub>oxide</sub>+P<sub>2</sub>O<sub>5</sub> recovery were also insignificant. For the **Non Magnetic Non Conductor** (Product 6) and the **Magnetic Non Conductor** (Product 7), the quartz contamination is the main problem (more than 50% by weight), and it is necessary a complementary stage of beneficiation by gravity separation (SCHNELLRATH et al., 2001). The principal mineral concentrated in these steps was the zircon. The **Non Magnetic Non Conductor** (Product 6) presented chemical composition of 1.8% TiO<sub>2</sub>, 8.3% ZrO<sub>2</sub> and 0.1% REE<sub>oxide</sub>+P<sub>2</sub>O<sub>5</sub>, and **Magnetic Non Conductor** (Product 7) of 2.6% TiO<sub>2</sub>, 11.8% ZrO<sub>2</sub> and 0.2% REE<sub>oxide</sub>+P<sub>2</sub>O<sub>5</sub>.

## 5. Conclusions

The sample 2 is composed of 10.14% of HM. Its mineralogical composition comprises magnetite, goethite, ilmenite, pseudorutile, staurolite, monazite, actinolite, albite, epidote, xenotime, zircon, kyanite, sillimanite, rutile,

anatase, quartz, muscovite, other silicates, amphiboles and feldspars, and the fraction with the greatest heavy mineral content was -74+53  $\mu\text{m}$ . Ilmenite is the principal Ti-bearing mineral and it is highly altered, mainly to pseudorutile and/or anatase. After the beneficiation tests, it was not obtained the individual specimens of heavy minerals of interest, being the Products 2 and 3 the fractions with the higher heavy mineral content. The quartz contamination after the electrostatic separation demonstrated the necessity of a complementary stage of beneficiation by gravity separation.

## 6. Acknowledgments

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