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Illegal Trade of Misclassified "Coltan" Concentrates in the Northwestern Amazonian Craton: Mineralogical Characterization and Radiological Screening of NORM-Bearing Mineral Concentrates

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Abstract

Illegal mining and the transboundary trade of poorly characterized mineral concentrates have expanded throughout the northwestern Amazonian Craton, particularly in eastern Colombia, southern Venezuela, and adjacent parts of northern Brazil. Materials marketed as "coltan" or "black sands" are commonly extracted, transported, and commercialized without adequate mineralogical characterization or radiological controls, despite the potential presence of naturally occurring radioactive materials (NORM). This study presents an integrated mineralogical, geochemical, and radiological screening assessment of confiscated mineral concentrates recovered from the Guainía and Vichada departments of eastern Colombia. A total of 24 samples, including black sands, wolframite-group minerals, iron oxides, and Nb-Ta-bearing concentrates, were investigated using field mineralogical observations, X-ray diffraction (XRD), wavelength-dispersive X-ray fluorescence (WDXRF), and gamma dose-rate measurements. The results demonstrate that materials traded as "coltan" or "black sands" are mineralogically heterogeneous and do not correspond predominantly to columbite–tantalite sensu stricto. Instead, Nb and Ta are primarily associated with Nb-rich rutile (ilmenorutile) and Ta-rich rutile (strüverite), whereas rare earth elements (REE), uranium, and thorium occur mainly within monazite–xenotime assemblages. Black-sand concentrates contain significant enrichments in REEs and NORM, with uranium contents reaching approximately 0.29 wt.% and thorium up to 0.57 wt.%. Radiometric measurements revealed a marked contrast between local background radiation and several confiscated concentrates. Background radiation averaged approximately 0.089 μSv/h under field conditions and ranged from 0.107 to 0.173 μSv/h during laboratory analyses, whereas storage areas containing confiscated materials reached approximately 4 μSv/h. Direct measurements above monazite-rich concentrates exceeded 20 μSv/h and reached up to 70 μSv/h in samples provided by indigenous communities. These findings indicate that a significant proportion of materials circulating through illegal mineral supply chains are misclassified and may contain elevated concentrations of NORM-bearing minerals. Although this study represents a preliminary radiological screening assessment rather than a comprehensive risk evaluation, the measured dose rates highlight the need for improved mineralogical characterization, radiological monitoring, and handling protocols for miners, traders, local communities, and law-enforcement personnel involved in the extraction, transport, storage, and confiscation of these materials. The results further demonstrate that illegally traded heavy-mineral concentrates from the northwestern Amazonian Craton constitute an underrecognized source of potential NORM exposure and emphasize

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the importance of integrating mineralogical verification and radiological screening into critical-mineral governance frameworks throughout northern South America.

Keywords: illegal mining; heavy-mineral concentrates; naturally occurring radioactive materials (NORM); rare earth elements (REE); radiological screening

1. Introduction

Illegal mining is increasingly recognized as a major driver of environmental degradation and pollution, particularly in regions where mineral extraction, processing, transportation, and storage occur outside environmental and mining regulatory frameworks. These activities are commonly associated with deforestation, sediment remobilization, contamination of soils and water resources, and uncontrolled human exposure to hazardous substances, including potentially toxic elements and naturally occurring radioactive materials (NORM) [1–3]. In tropical environments, intense chemical weathering and prolonged erosion promote the residual enrichment and hydraulic concentration of dense, chemically resistant minerals within alluvial deposits and lateritic profiles [4,5], making heavy-mineral sands an important but often overlooked source of NORM-bearing mineral concentrations and potential radiological hazards.

The environmental significance of NORM-bearing heavy-mineral deposits has been documented in several regions worldwide. Monazite-, xenotime-, zircon-, rutile-, and ilmenite-rich placers in Brazil, Sri Lanka, India, and Australia contain elevated concentrations of uranium and thorium, generating enhanced natural gamma radiation and requiring radiological monitoring during mining and mineral processing [6,7]. These studies demonstrate that heavy-mineral concentrates can constitute important sources of NORM occurrence and potential occupational exposure when extracted, transported, processed, or stored without appropriate radiological controls [8,9]. Nevertheless, comparable investigations remain scarce in tropical regions affected by informal and illegal mining, particularly within the Amazonian and Orinoquian regions of northern South America.

Eastern Colombia, encompassing the Amazonian and Orinoquian regions (Figure 1), has recently emerged as an area of growing environmental concern due to the rapid expansion of unregulated extraction and commercialization of materials locally referred to as “coltan” [10,11]. Similar mining activities occur throughout the northwestern Amazonian craton, including southern Venezuela and northern Brazil, where limited environmental oversight, political instability, and transboundary trafficking facilitate the circulation of poorly characterized mineral concentrates across national borders. In Colombia, these activities concentrate along the Inírida, Guainía, and Orinoco river systems and often occur within protected ecosystems and Indigenous territories. Despite increasing governmental enforcement efforts, the mineralogical, geochemical, and radiological characteristics of these materials remain poorly understood, limiting robust assessments of their mineralogical composition and radiological significance.

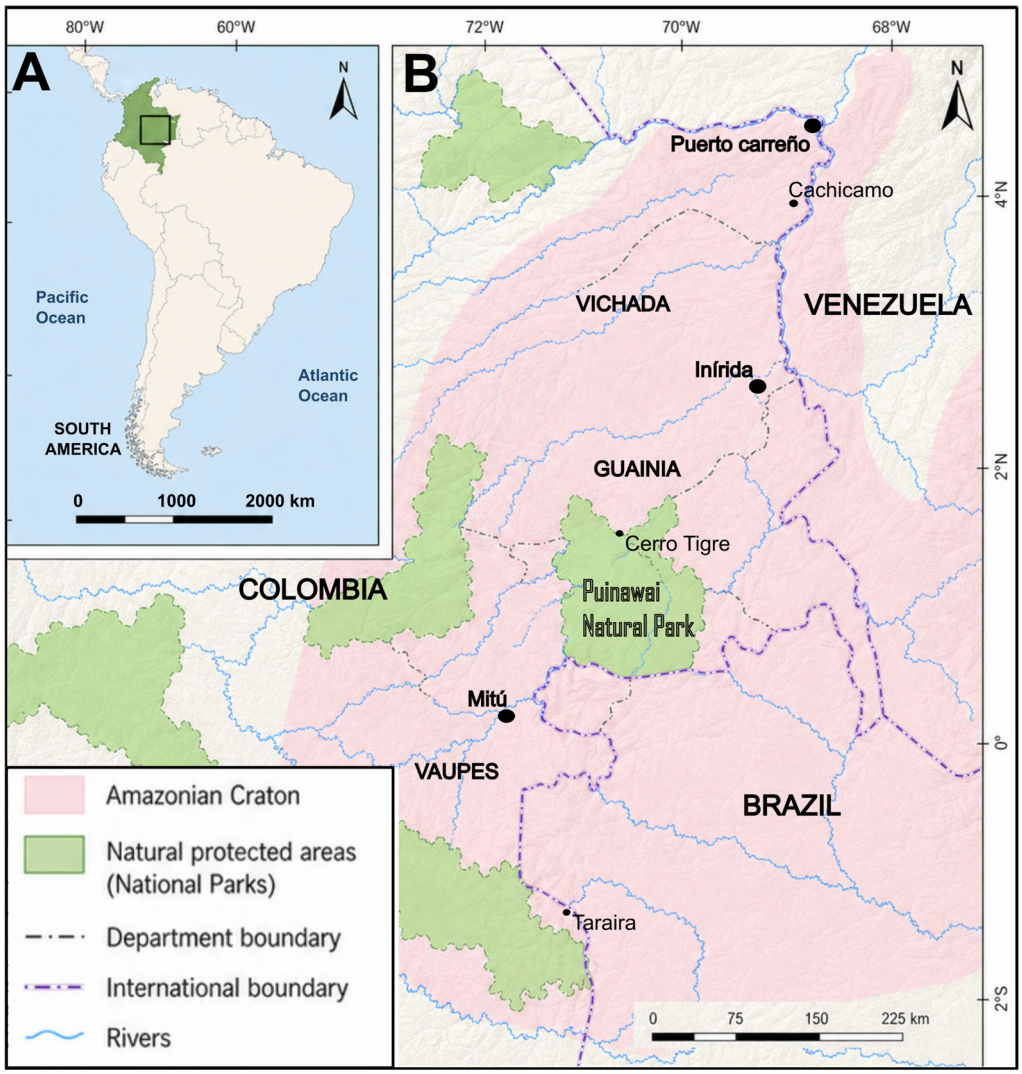


Figure 1. (**A**) Location of Colombia in northern South America; (**B**) the study area within eastern Colombia and the northwestern Amazonian Craton in northern South America and some known ore occurrences and deposits, such as Taraira (gold, wolframite), Cerro Tigre (wolframite), and Cachicamo (ilmenorutile).

Geologically, eastern Colombia forms part of the northwestern Amazonian Craton (Figure 1), a Precambrian terrane that experienced multiple Paleoproterozoic and Mesoproterozoic tectono-magmatic events [12–19] that enriched it in high-field-strength elements (HFSE) and rare earth elements (REE) [10,11,20–22]. Mesoproterozoic A-type granites, including the Parguaza Granite and related intrusive suites [18,19], constitute major sources of Nb–Ta–Sn–W–REE–U–Th mineralization (Figure 2A). Weathering-resistant accessory minerals such as ilmenorutile, strüverite, monazite, xenotime, zircon, and wolframite survive prolonged tropical weathering and become progressively concentrated within alluvial placers and lateritic deposits through hydraulic sorting (Figure 2B) [4], creating economically attractive heavy-mineral concentrations that are readily exploited by artisanal and illegal miners (Figure 2C,D).

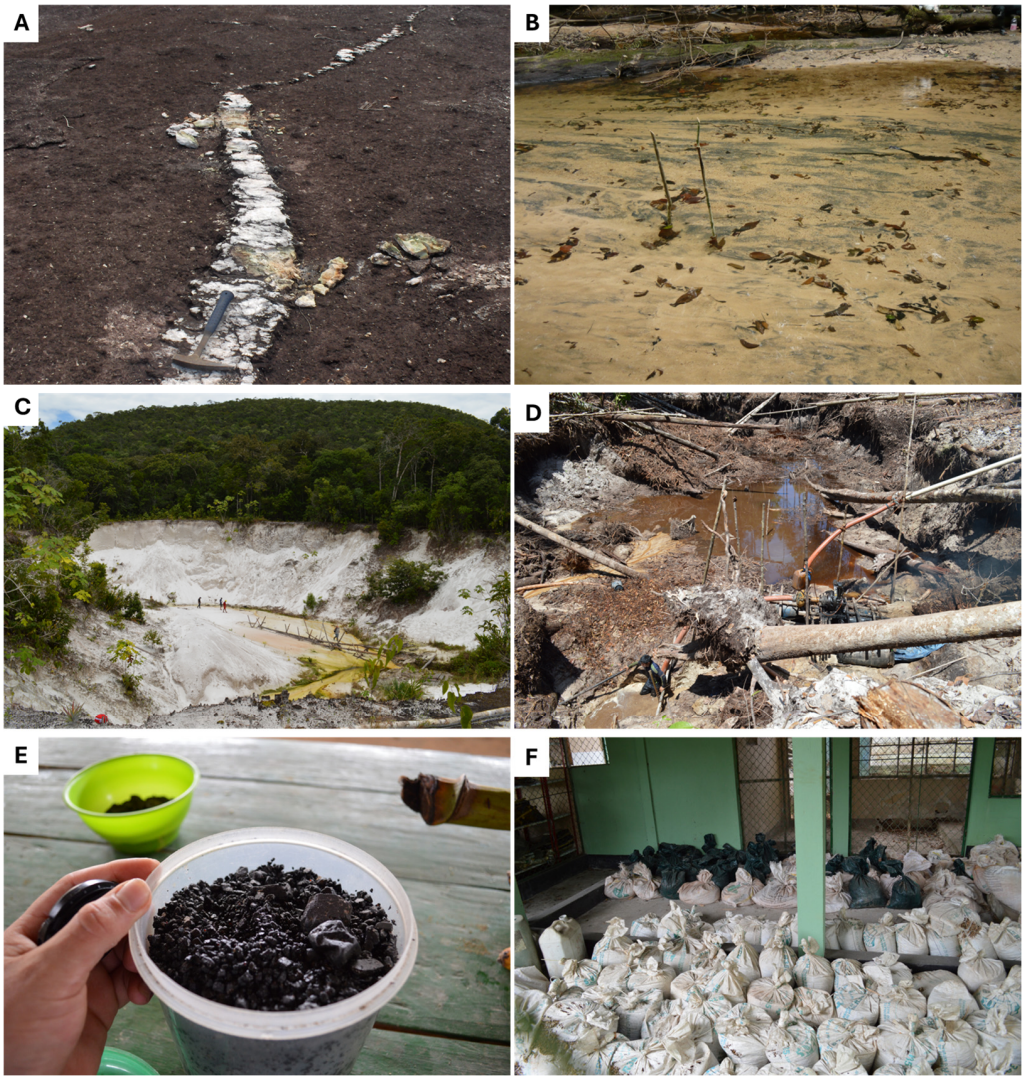


Figure 2. Mining process chain and stages relevant to mineral concentration and NORM occurrence in eastern Colombia. (**A**) Precambrian basement rocks intruded by pegmatitic dikes, representing the primary geological source of Nb–Ta–REE–W-bearing minerals in the Guainía Department. (**B**) Active fluvial systems where weathering and hydraulic sorting concentrate heavy minerals within black-sand deposits in the Vichada Department. (**C**,**D**) Unregulated extraction of alluvial and colluvial deposits, illustrating illegal mining activities that disturb sediments and riverbanks and promote uncontrolled mobilization of mineral concentrates in the Guainía Department. (**E**) Accumulated heavy-mineral concentrates locally and incorrectly referred to as "coltan," obtained from alluvial extraction without the use of personal protective equipment or environmental controls, posing potential exposure risks to indigenous and local communities. (**F**) Confiscated mineral materials stored by the Policía Nacional under inadequate safety conditions, without prior mineralogical characterization or radiological protection measures, represent a potential source of occupational exposure and radiological concern.

A critical environmental issue arises from the widespread misclassification of these mineral concentrates. In Colombia, the commercial term "coltan" is broadly applied to almost any dense, dark-colored heavy-mineral concentrate (Figure 2E), irrespective of its true mineralogical composition. This commercial misnomer promotes indiscriminate extraction and trade of heterogeneous mineral assemblages that often contain economically valuable critical minerals alongside significant concentrations of uranium- and thorium-bearing phases. Consequently, materials marketed as "coltan" commonly comprise assemblages dominated by Nb-rich rutile (ilmenorutile), Ta-rich rutile (strüverite), monazite, xenotime,

zircon, iron oxides, and wolframite, rather than true columbite–tantalite minerals. Monazite and xenotime are recognized worldwide as the principal hosts of REE, uranium, and thorium in heavy-mineral deposits and therefore constitute important NORM-bearing minerals [23,24] that can generate elevated external radiation levels and elevated concentrations of naturally occurring radioactive materials when handled without appropriate controls [25,26].

Beyond conventional geochemical pollution, illegal exploitation of these materials introduces an often-overlooked radiological dimension to illegal mineral exploitation. The concentration, storage, transportation, and commercialization of NORM-bearing mineral concentrates can generate elevated gamma radiation fields while increasing the potential for inhalation or ingestion of radioactive mineral particles during mining, crushing, handling, and storage (Figure 2F) [3,25,26]. These exposure pathways are particularly relevant in tropical regions where mining is commonly undertaken by Indigenous communities and artisanal miners under limited technical supervision and minimal environmental regulation. Unlike industrial mineral-sands operations in Australia or Brazil, where radiological protection protocols are routinely implemented, illegal mining activities in northern South America generally lack systematic monitoring, exposure assessment, and environmental management.

In this study, illegal mining refers to the extraction, handling, transportation, storage, and commercialization of mineral resources outside established environmental, radiological, and mining regulatory frameworks. This definition emphasizes the lack of mineralogical characterization, traceability, and control measures, which may result in the circulation of poorly characterized heavy-mineral concentrates containing naturally occurring radioactive materials (NORM), and is consistent with recent approaches used to evaluate environmental and societal impacts associated with informal mining activities [1–3,6–9,25,26]. The present study provides an integrated mineralogical, geochemical, and radiological screening assessment of confiscated heavy-mineral concentrates from eastern Colombia that are commercially traded as “coltan”. Specifically, the objectives are to: (i) identify the mineral phases hosting Nb, Ta, rare earth elements (REE), uranium, and thorium; (ii) determine the mineralogical controls governing the distribution of critical and radioactive elements within these concentrates; (iii) quantify gamma dose rates associated with the analyzed materials; and (iv) evaluate the implications of their uncontrolled extraction, transport, storage, and commercialization for potential radiological exposure along illegal mineral supply chains. To our knowledge, this is the first integrated mineralogical, geochemical, and radiological investigation of illegally traded Nb-Ta-REE-bearing heavy-mineral concentrates from the northwestern Amazonian Craton. By linking mineralogical composition with measured radiation levels, this study demonstrates that materials marketed as “coltan” are frequently misclassified and may contain significant concentrations of NORM-bearing minerals. The results provide a scientific basis for improving mineral identification, radiological screening, regulatory oversight, and risk-management strategies for miners, traders, local communities, and law-enforcement personnel handling these materials.

2. Materials and Methods

2.1. Sample Collection

Twenty-four representative mineral samples were collected during field campaigns conducted between January and July 2019 in the departments of Guainía and Vichada, eastern Colombia. Because the objective of this study was to evaluate the environmental and radiological hazards associated with mineral materials circulating through illegal mining networks rather than to characterize primary ore bodies, sampling focused on mineral concentrates and extracted materials obtained from two complementary sources.

The first group of samples was acquired directly from miners operating along the Inírida and Orinoco river systems, where heavy-mineral concentrates are commonly recovered from alluvial deposits. Owing to the illegal nature of these activities and the need to protect the identity of the miners, precise sampling coordinates and extraction sites were not recorded. This limitation does not affect the study's main objective: to characterize the mineralogical composition and radiological properties of the materials handled, transported, stored, and traded within the illegal supply chain.

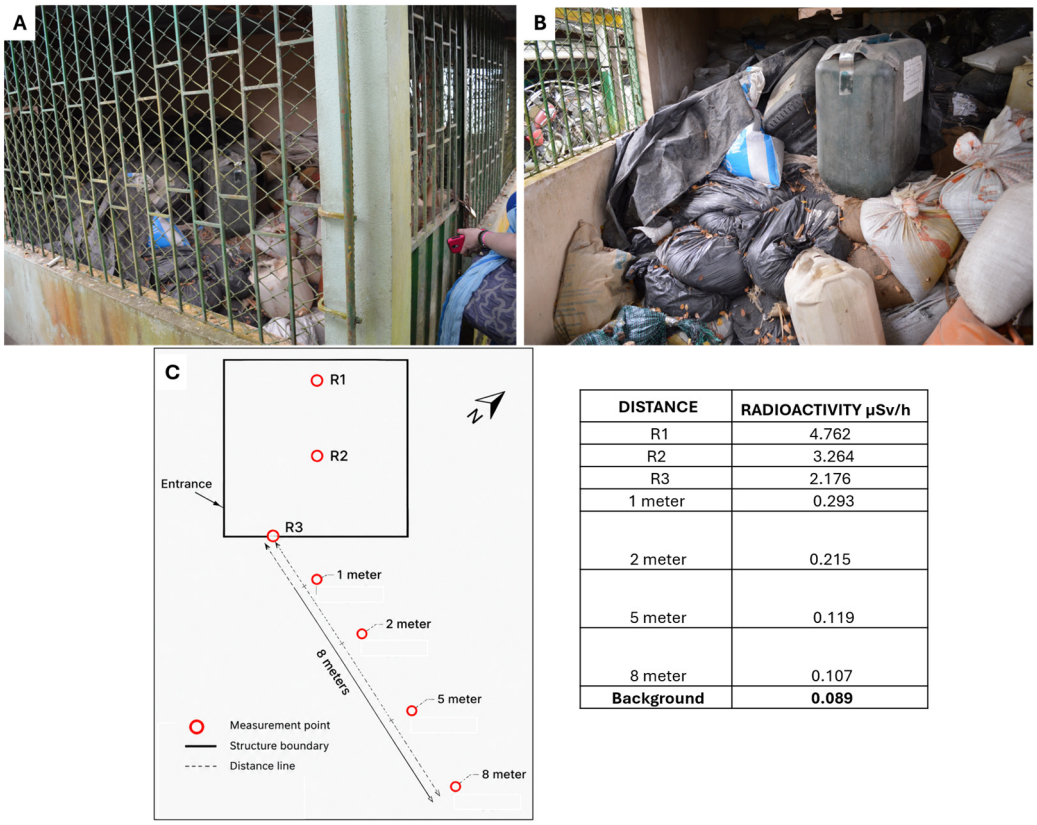
The second group consisted of confiscated mineral materials provided by the Colombian National Police in Inírida (Guainía) and Puerto Carreño (Vichada) (Figure 2E,F). Law enforcement had previously seized these materials during operations against illegal mining and trafficking of mineral concentrates.

Sampling involved randomly selecting representative sub-samples from heterogeneous bags or piles of confiscated concentrates. Approximately 1 kg of material was collected for each sample. The sampled materials included black sands, gravels, and nodule concentrates. Macroscopic characterization included color, luster, streak, magnetic response, relative density, and grain morphology following standard mineral identification procedures [27]. Immediately after collection, each sample was placed in cylindrical plastic containers internally shielded with approximately 0.5 cm of lead for transportation to the laboratories in Bogotá. Following arrival, all samples were stored inside a 3 cm thick lead-shielded storage container to minimize radiation exposure. Approximately 50 g of each sample was subsequently subsampled for mineralogical and geochemical analyses. All sample metadata are recorded in the Supplementary Materials.

2.2. Radioactivity

Gamma-radiation measurements were performed using a portable InspectorAlert™ radiation survey meter (International Medcom, Inc., Sebastopol, CA, USA), equipped with a halogen-quenched Geiger–Müller detector for measuring gamma radiation. The manufacturer supplied the instrument with a Certificate of Conformance and standard factory calibration at the time of purchase (2018). According to the manufacturer, the instrument was electronically calibrated to factory specifications using a pulse generator, with an accuracy typically within ±15% relative to a 137 Cs reference source. Gamma dose rates were measured both on individual mineral samples and in storage areas containing confiscated mineral concentrates. Measurements on individual samples were performed by positioning the detector approximately 5 cm above the sample surface. Count times continued until readings stabilized, typically after about 30 s, and dose rates were recorded in μSv/h. Ambient background radiation was measured periodically during laboratory measurements using a counting interval of approximately 60 s with laboratory background values ranged from 0.107 to 0.173 μSv/h. These background measurements are reported in the Supplementary Materials.

Additional measurements were conducted at increasing distances from stored mineral concentrates (Figure 3C) to evaluate gamma-radiation attenuation and assess potential exposure scenarios associated with storage and handling. Measurements obtained directly above mineral samples and those acquired within storage facilities were treated as separate exposure scenarios, allowing distinction between direct contact with concentrates and ambient exposure within storage environments with a field background radiation averaged 0.089 μSv/h.



DISTANCE	RADIOACTIVITY µSv/h
R1	4.762
R2	3.264
R3	2.176
1 meter	0.293
2 meter	0.215
5 meter	0.119
8 meter	0.107
Background	**0.089**

Figure 3. Storage conditions of confiscated mineral materials at Warehouse No. 1. (**A**) Exterior view of Warehouse No. 1, used for the storage of mineral materials confiscated by national authorities. (**B**) Interior view illustrating inadequate storage conditions, including the mixing of heterogeneous mineral materials in bags and containers, lack of segregation, and absence of protective measures, which increase the potential for uncontrolled handling and potential radiological exposure. (**C**) Schematic storage conditions showing the radioactivity measurements by distance.

The objective of the radiometric measurements was not to conduct a comprehensive radiological risk assessment, but to identify naturally occurring radioactive materials (NORM) in illegally traded heavy-mineral concentrates and to provide a preliminary radiological screening of materials circulating through illegal mineral supply chains. Measured dose rates were therefore evaluated relative to local background radiation levels and interpreted as indicators of radiological significance that may warrant further investigation.

2.3. Wavelength-Dispersive X-Ray Fluorescence (WDXRF)

Major and trace element compositions were determined using a wavelength-dispersive X-ray fluorescence (WDXRF) spectrometer (PANalytical Axios), equipped with a 4 kW Rhodium X-ray tube operating at 60 kV and 160 mA. Analytical procedures included preparing fused glass beads for major-element determinations and pressed powder pellets for trace-element determinations, following standard protocols to ensure accuracy and reproducibility. The WDXRF data were used to quantify major oxides such as WO_3, Fe_2O_3, MnO, TiO_2, Nb_2O_5, and Ta_2O_5, which are critical for distinguishing between wolframite-group minerals (ferberite–hübnerite series), Nb–Ta oxides, and Ti-bearing phases. These geochemical signatures are essential for mineral classification, provenance analysis, and identifying misclassified materials (e.g., “coltan”). Fe is reported as Fe_2O_3 (total iron) from XRF; however, for classification purposes, it is treated as FeO equivalent.

In line with previous studies on alkaline and rare-element mineral systems in Colombia, particular attention was given to elements associated with rare-earth element (REE)

mineralization and high-field-strength elements (HFSE), which are key indicators of magmatic and hydrothermal processes and may also reflect the economic potential of these materials. Elements below detection limits (~50 ppm) may not be detected. The analysis corresponds to a fully quantitative program.

2.4. X-Ray Diffraction (XRD)

Mineralogical characterization was carried out using a PANalytical X'Pert Pro MPD diffractometer operating in θ–θ configuration, equipped with a high-speed solid-state detector and a monochromator to reduce fluorescence effects, particularly in Fe-rich samples.

XRD analyses identified crystalline phases in the samples, including wolframite-group minerals, ilmenite, rutile, hematite, magnetite, monazite, xenotime, and other accessory phases. The use of a monochromator was particularly important for improving signal quality in samples with high iron content and complex mineral assemblages.

3. Results

Mineralogical interpretations presented in this study are based on an integrated analytical approach combining XRD, WDXRF, and field mineralogical observations. Minerals reported as "identified" were directly recognized by XRD analysis. Minerals marked as inferred (*) were interpreted from geochemical signatures obtained by XRF together with mineral associations recognized in XRD patterns. Analyses demonstrate that materials marketed as "coltan" comprise are mineralogically diverse and consist of three principal groups: (i) wolframite group minerals, (ii) black sand–gravel assemblages dominated by Ti, Nb, and Ta-bearing oxides and phosphates with REE-bearing, and (iii) iron oxides and hydroxides.

3.1. Wolframite Group Minerals (Tungsten)

Of the 24 collected samples, five were selected based on macroscopic identification as wolframite-group minerals. These samples were subjected to (WDXRF) analysis to quantify their chemical composition, and the results clearly indicate that all samples (Table 1) correspond to wolframite-group minerals, with tungsten (WO_3) contents exceeding 65 wt.%. Although anomalous concentrations of niobium and tantalum are detected, these values are not considered geochemically significant.

Elevated bismuth concentrations are observed in association with the wolframite samples. Such anomalies may indicate mineralization processes associated with gold-bearing systems. However, the samples analyzed showed no significant gold anomalies (Table 1).

Sample 1.10 exhibits notable concentrations of rare earth elements (REE), including cerium and yttrium, as well as radioactive elements such as uranium, at levels of potential economic interest. These results are consistent with the elevated radioactivity values measured for this sample, in contrast to the other wolframite samples. The presence of these elements suggests accessory REE-bearing minerals, likely monazite, which commonly forms under geological conditions associated with wolframite mineralization. X-ray diffraction (XRD) analysis was conducted to identify and differentiate the mineral phases present in each sample. A key limitation arises from the strong similarity between the diffraction patterns of hübnerite and ferberite. Their diffractograms are closely comparable, making it difficult to distinguish between these mineral varieties based solely on XRD data (Figure 4).

Table 1. Major-element chemical composition (wt.%) of samples identified as wolframite by XRF analysis.

Sample	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	K_2O	P_2O_5	ZrO_2	ZnO	Y_2O_3	Nb_2O_5	CeO_2	Ta_2O_5	Bi_2O_3	WO_3	U
Sample 1.4 Gravel	0.91	b.d.l	0.59	6.29	17.33	b.d.l.	b.d.l	b.d.l	b.d.l	b.d.l	0.12	b.d.l	b.d.l	b.d.l	74.73	b.d.l
Sample 1.5 Gravel	4.6	b.d.l	3.0	9.2	14.8	0.3	b.d.l	b.d.l	0.1	b.d.l	0.2	b.d.l	b.d.l	0.2	67.7	b.d.l
Sample 1.10 Red	1.6	1.9	0.8	11.3	15.2	0.1	0.1	0.1	0.1	0.2	0.5	1.2	0.3	0.1	66.5	0.1
Sample 4 Gravel	3.2	b.d.l	3.3	9.4	15.2	0.1	b.d.l	b.d.l	b.d.l	b.d.l	0.2	b.d.l	b.d.l	0.2	68.5	b.d.l
Sample 5 Gravel	3.1	b.d.l	1.3	8.1	16.7	b.d.l	b.d.l	b.d.l	b.d.l	b.d.l	0.2	b.d.l	b.d.l	0.1	70.5	b.d.l

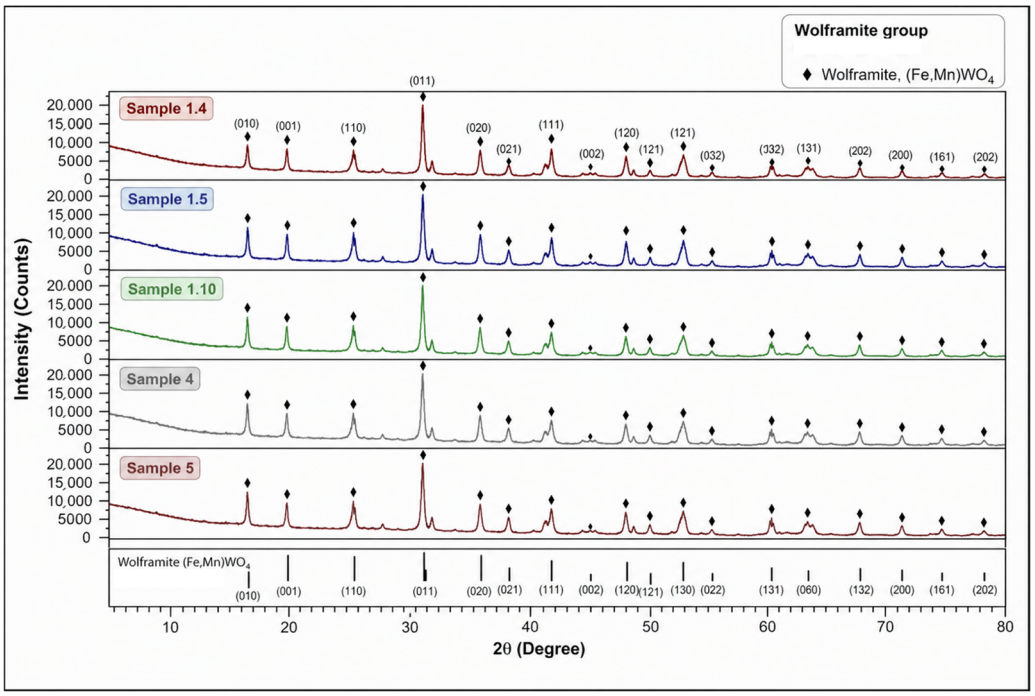


Figure 4. X-ray diffraction (XRD) patterns of the analyzed wolframite samples (Samples 1.4, 1.5, 1.10, 4, and 5). Note the strong similarity among diffractograms, reflecting comparable crystalline structures within the wolframite group.

However, integrating XRD results with geochemical analyses and considering the compositional definitions of these minerals—hübnerite (MnO-dominant) and ferberite (FeO-dominant)—refines the classification. Based on this combined approach, the analyzed samples are interpreted as predominantly hübnerite (MnO > FeOt) (Tables 1 and 2).

Table 2. Mineral phases identified in wolframite samples.

Sample	Main Phase	Accessory Phases
Sample 1.4	Hübnerite	Zircon
Sample 1.5	Hübnerite	Quartz
Sample 1.10	Hübnerite	Quartz, Hematite, Monazite *, Zircon *
Sample 4	Hübnerite	Quartz
Sample 5	Hübnerite	Quartz

* Accessory minerals inferred from geochemical data.

3.2. Black Sands-Gravels

Sand is defined as a collection of disaggregated rock particles with grain sizes ranging from 0.063 to 2 mm; gravel has grain sizes > 2 mm. In continental and non-tropical coastal settings, quartz is typically the dominant mineral due to its resistance to weathering. However, their composition varies depending on local geological conditions and source rocks. In certain regions, those sediments naturally contain significant proportions of iron oxides, titanium oxides, and other dense, dark-colored minerals.

A total of 14 samples identified as black sands-gravels (primarily composed of ilmenite, ilmenorutile, rutile, zircon, and monazite) were selected for XRF analysis to quantify their chemical composition (Supplementary Materials). For Sample 1.3, a manual separation was performed to separate black grains from brown grains to obtain individual compositional results. Niobium (Nb) and tantalum (Ta) are the principal elements in materials colloquially referred to as "coltan." In the samples analyzed, Nb concentrations reach up to 15 wt.% and Ta up to 8 wt.%. Samples showing significant values of these elements include 1.3,

1.6, 1.7, 1.8, 1.9, and 10 (e.g., Figure 5 and Supplementary Materials). However, the mineralogical context indicates that these materials contain high concentrations of titanium and iron, suggesting that they do not correspond to minerals of the columbite–tantalite series, which are the true sources of "coltan." Instead, the geochemical composition reveals clear differences among the sampled materials and, importantly, unexpected results in terms of elevated concentrations of rare earth elements (REE), including cerium (Ce), yttrium (Y), lanthanum (La), and ytterbium (Yb), as well as radioactive elements such as uranium (U) and thorium (Th). These elements are primarily concentrated in monazite, which, due to its high specific gravity, accumulates with other heavy minerals, such as ilmenite, rutile, tantalum-bearing oxides, columbite, cassiterite, and related phases, within black sand deposits.

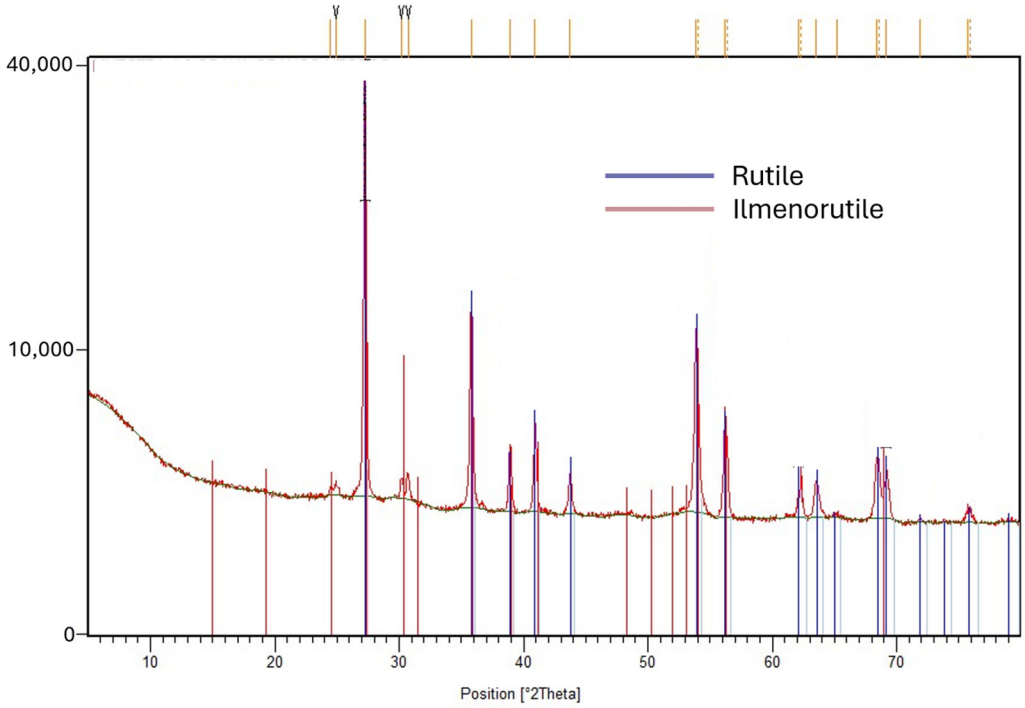


Figure 5. X-ray diffraction (XRD) patterns of the analyzed black sand and gravel (Samples 1.3A black grains).

XRD analyses did not detect minerals belonging to the columbite–tantalite series among the analyzed samples (i.e., "coltan") as primary or accessory phases (Supplementary Materials). Instead, the elevated Nb and Ta concentrations are closely associated with titanium-oxide-rich assemblages, particularly Nb-rich rutile (ilmenorutile) and Ta-rich rutile (strüverite). Confirmation of elemental partitioning within individual mineral grains requires future SEM-EDS or EPMA analyses, specifically Nb-rich rutile (Nb-rutile) and Ta-rich strüverite (Ta-rutile), respectively (Table 3). Rare-earth elements are hosted in monazite, which occurs in minor amounts in some samples (e.g., 4.2, 6, 7, 8, and 10) but in significant proportions in others, reaching up to 20 vol.% in Sample 1.3B (Supplementary Materials).

Sample 10 exhibits a distinct mineralogical assemblage compared to the other samples, including tungsten-bearing minerals such as hübnerite. This may reflect genetic differences or suggest mixing of materials from different sources, possibly related to commercial handling or illegal trade practices (Table 3).

Table 3. Mineralogical phases identified in black sand and gravel samples.

Sample	Main Phase	Accessory Phases
Sample 1	Ilmenite, Rutile, Hematite	Quartz, Monazite
Sample 1.1	Ilmenite, Rutile, Hematite	Quartz, Monazite
Sample 1.3	Nb-rutile (ilmenorutile), Ta-Rutile(strüverite), Rutile, Monazite, Xenotime	Quartz
Sample 1.6	Nb-rutile (ilmenorutile), Ta-rutile (strüverite)	Quartz, Monazite
Sample 1.7	Nb-rutile (ilmenorutile), Ta-rutile (strüverite), Rutile	
Sample 1.8	Ilmenite, Hematite	Monazite, Quartz, Zircon
Sample 1.9	Nb-rutile (ilmenorutile), Ta-rutile (strüverite), Ilmenite	Quartz, Monazite, Zircon *
Sample 3	Ilmenite, Hematite	Quartz
Sample 4.2	Ilmenite, Hematite	Quartz, Monazite *
Sample 6	Ilmenite, Hematite	Quartz, Monazite *
Sample 7	Ilmenite, Hematite	Quartz
Sample 8	Ilmenite	Quartz, Monazite *
Sample 10	Ilmenite, Hübnerite, Nb-Rutile *	Monazite *

* Accessory minerals inferred from geochemical analyses.

3.3. Iron Oxides and Hydroxides

Five samples were identified as iron oxides and hydroxides, primarily composed of hematite and limonite nodules, with some containing quartz and ilmenite grains. These materials are readily distinguishable from other sample types based on their relatively low density, morphology, weak or absent magnetic response, and characteristic reddish color upon fracture.

Four samples were selected for X-ray fluorescence (XRF) analysis to quantify their chemical composition (Table 4). Sample 1.11 was specifically identified as hematite of the specularite variety. The analytical results indicate that these samples do not exhibit anomalous concentrations of elements of interest such as Nb, Ta, Sn, or Ti, so DRX was not applied. Instead, they correspond predominantly to iron- and silica-rich materials. Most samples were moist, as reflected in relatively high loss-on-ignition (LOI) values, up to 10 wt.% (e.g., Sample 4.1).

Table 4. Major element composition (wt.%) of samples dominated by quartz and iron oxides/hydroxides (LOI: Loss on ignition), determined by XRF analysis.

Sample	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	K_2O	P_2O_5	V_2O_5	Cr_2O_3	ZrO_2	LOI
Sample 2 gravel	97.64	0.12	0.54	0.76	b.d.l	0.09	b.d.l	b.d.l	b.d.l	b.d.l	0.80
Sample 2.1 gravel	16.55	0.52	12.57	61.23	b.d.l	0.16	b.d.l	0.25	0.05	b.d.l	8.66
Sample 4.1 gravel	17.06	0.52	12.50	59.37	b.d.l	0.17	b.d.l	0.25	0.05	b.d.l	9.12
Sample 9 gravel	40.76	0.66	18.14	29.25	0.03	0.34	0.03	0.06	b.d.l	0.06	10.75
Sample 11	16.13	0.48	8.36	67.86	b.d.l	0.14	b.d.l	0.28	0.03	0.03	6.73

Sample 2 is distinguished by its high SiO_2 content, consisting mainly of quartz (>80 wt.%) with minor clay components. Meanwhile, samples 2.1, 4.1, 9, and 11 correspond to iron oxides, primarily hematite (Table 5), as well as iron and aluminum hydroxides and silicate phases such as goethite and kaolinite. Significant amounts of limonite are also present, interpreted as alteration products of hematite. The low titanium concentrations indicate that these samples lack Ti-bearing minerals such as ilmenite or rutile.

Table 5. Mineralogical phases identified in nodule samples.

Sample	Main Phase	Accessory Phases
Sample 1.11	Hematite	n.a.
Sample 2	Quartz	Clays
Sample 2.1	Hematite, Kaolinite	Limonite, Goethite
Sample 4.1	Hematite, Kaolinite	Limonite, Goethite
Sample 9	Quartz, Hematite, Kaolinite	Limonite, Goethite
Sample 11	Hematite, Kaolinite	Limonite, Goethite

3.4. Nb-Rich Rutile and Ta-Rich Rutile

The best-known occurrence of tantalum and niobium minerals in eastern Colombia is at Cachicamo (Vichada). The dominant material is ferruginous gravel, characterized by poor sorting and irregular grains. The deposit is interpreted as a combination of colluvial material, lateritic profiles (incomplete lateritic profile), and exposed basement. Metallic mineral fragments of economic interest, up to 2 cm in size (Nb-rich rutile and strüverite ± cassiterite–columbite), occur within porous and irregular aggregates cemented by iron oxides.

X-ray fluorescence (XRF) analyses were performed on rutile grains enriched in tantalum and niobium from this locality. The results indicate a composition of approximately 52 wt.% TiO_2, 15 wt.% Ta_2O_5, 15 wt.% Nb_2O_5, 10 wt.% Fe_2O_3, and 3 wt.% SnO_2. Meanwhile, X-ray diffraction (XRD) analysis was carried out on Sample #5 from the Cachicamo deposit. The results confirm the presence of titanium oxides enriched in niobium and tantalum (Nb-rich rutile and Ta-rich rutile). This rutile is not compositionally homogeneous and contains inclusions of tin oxides (cassiterite) and niobium–tantalum oxides (ferrocolumbite) (Figure 6 and Table 6).

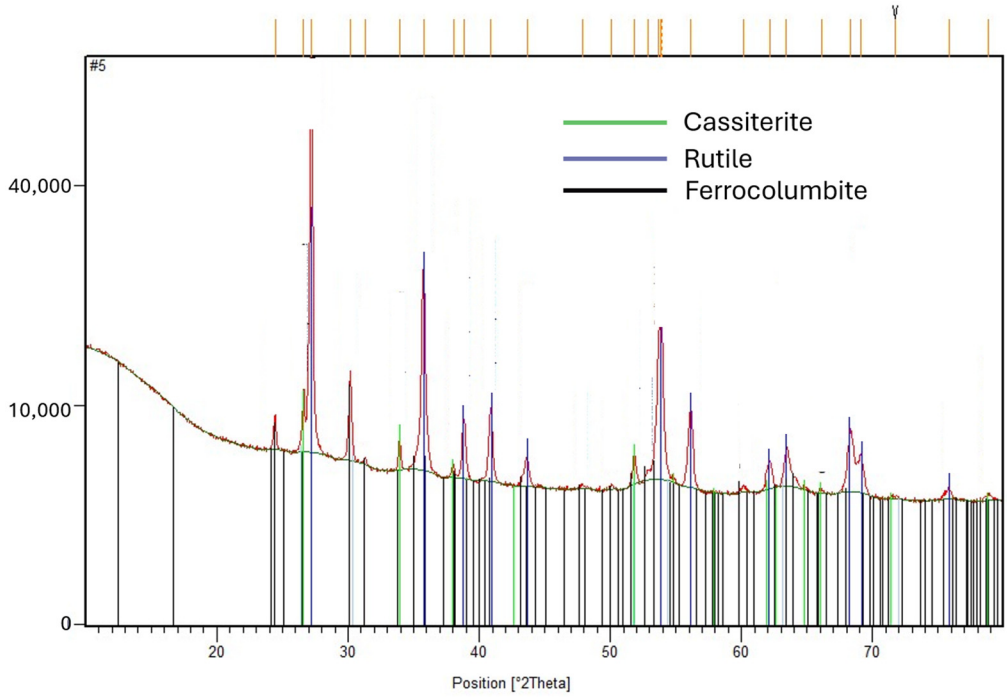


Figure 6. X-ray diffraction (XRD) patterns of the analyzed sample from Cachicamo (Sample 5).

Table 6. Major element composition (wt.%) of Nb- and Ta-bearing rutile (including strüverite), based on XRF analysis.

Sample	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	P_2O_5	ZrO_2	Nb_2O_5	SnO_2	Ta_2O_5	WO_3
#5	0.29	52.95	0.22	10.11	0.46	0.03	0.19	15.62	3.97	15.65	0.51

Elevated concentrations of Fe_2O_3, Al_2O_3, and SiO_2 (Table 7) indicate the presence of iron oxides, including magnetite, hematite, and ilmenite, which are also identified as mineral phases in the XRD patterns (Supplementary Materials). Other clay minerals, including kaolinite, illite, and gibbsite, require more specialized analytical procedures for definitive XRD identification. However, their presence can be reasonably inferred based on geochemical composition and the occurrence of quartz grains, which are also observed macroscopically.

Table 7. Major element composition (wt.%) of iron oxide/hydroxide samples from the Cachicamo deposit (Vichada), based on XRF analysis.

Sample	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	K_2O	P_2O_5	LOI	V_2O_5	ZrO_2	BaO	ZnO	Y_2O_3	Nb_2O_5
#3A	57.73	0.99	11.53	22.41	0.05	0.03	7.01	b.d.l	0.24	b.d.l	b.d.l	b.d.l	0.03
#4	1.55	4.70	4.26	87.06	0.31	0.39	b.d.l	0.18	0.10	0.03	0.39	0.1	b.d.l
#6	58.50	0.80	10.24	23.84	0.04	0.05	6.37	b.d.l	0.17	b.d.l	b.d.l	b.d.l	b.d.l

4. Discussion

4.1. Misclassification of "Coltan" in the Northwestern Amazonian Craton

One of the most significant findings of this study is that materials commercially traded as "coltan or black sands" do not correspond exclusively, and in many cases not predominantly, to minerals of the columbite–tantalite series [28]. Instead, confiscated concentrates from eastern Colombia (e.g., [29–31]) comprise a heterogeneous assemblage of heavy minerals including rutile-group Nb-Ta-bearing phases, wolframite-group minerals, monazite, xenotime, zircon, ilmenite, and iron oxides. This mineralogical diversity demonstrates that the commercial use of the term "coltan" substantially oversimplifies the actual composition of materials circulating through regional mineral supply chains, and the use of "black sands" could be more adequate for the mixed material traded. The XRD results indicate that Nb and Ta are commonly associated with rutile-group minerals, particularly ilmenorutile and strüverite, rather than with columbite–tantalite sensu stricto. Similar mineralogical associations have been documented in evolved pegmatitic and rare-metal systems where Nb and Ta may occur in a variety of oxide phases rather than exclusively within columbite-group minerals [20,28]. Although the XRD and WDXRF results consistently indicate a strong association between Nb-Ta enrichment and rutile-group minerals, the precise distribution of Nb and Ta within individual grains remains to be confirmed through microanalytical techniques such as SEM-EDS or EPMA.

From a resource perspective, these observations indicate that exploration programs focused solely on identifying columbite–tantalite may underestimate the critical-mineral potential of placer systems throughout the northwestern Amazonian Craton. More importantly, the results demonstrate that the widespread commercial classification of these materials as "coltan" (e.g., [29–31]) may obscure significant variations in mineralogical composition, economic value, and radiological characteristics.

4.2. Heavy-Mineral Assemblages and Placer Formation Processes

The identified mineral assemblages are consistent with mature polymetallic placer systems generated through prolonged weathering, erosion, transport, and hydraulic con-

centration. Rutile, zircon, monazite, xenotime, and wolframite exhibit high density and chemical stability, enabling their accumulation within alluvial environments after repeated cycles of sedimentary reworking [4,5,23–25]. The coexistence of rutile-group minerals with monazite, xenotime, and zircon suggests derivation from complex source rocks, such as pegmatites [29,32–34] containing multiple accessory mineral phases. Under tropical weathering conditions characteristic of the Amazonian and Orinoquian regions, chemical alteration preferentially removes unstable phases while preserving denser and more resistant minerals. Subsequent hydraulic sorting then concentrates these minerals within black-sand deposits and gravel-rich placer horizons. However, the marked mineralogical variability observed among confiscated concentrates suggests that materials marketed as "coltan" or "black sands" may contain mixtures derived from different sources. Such mixing could obscure the presence of REE-rich and NORM-bearing components within commercialized concentrates and complicate traceability throughout regional trade networks.

The occurrence of monazite and xenotime is particularly significant because these minerals represent major repositories of rare earth elements and have been reported in eastern Colombia [21]. Monazite typically hosts light rare earth elements and thorium, whereas xenotime commonly concentrates yttrium and heavy rare earth elements [22,24,25,35,36]. Consequently, the studied deposits should not be viewed solely as Nb-Ta occurrences but rather as polymetallic heavy-mineral systems containing a broader spectrum of critical elements (e.g., [26,37]). The wide mineralogical variability observed among confiscated samples suggests that materials entering regional supply chains may originate from multiple source areas within the northwestern Amazonian Craton, including eastern Colombia, southern Venezuela, and adjacent regions of northern Brazil. This diversity further complicates the use of generic commercial labels and highlights the need for systematic mineralogical verification of concentrates traded as "coltan".

4.3. NORM Occurrence and Mechanisms of Radioactive Mineral Enrichment

Several analyzed samples contain minerals widely recognized as hosts of naturally occurring radioactive materials (NORM), principally monazite, xenotime, and zircon. These minerals commonly incorporate variable proportions of uranium and thorium within their crystal structures and could represent important sources of natural radioactivity in heavy-mineral deposits worldwide (e.g., [6,25,35,38,39]). The observed NORM enrichment likely results from a combination of geological and anthropogenic processes. At the geological scale, uranium- and thorium-bearing accessory minerals formed within primary igneous, pegmatitic, or metamorphic source rocks and were subsequently liberated during weathering. Because monazite and xenotime are highly resistant to chemical alteration, they tend to accumulate progressively during sediment transport and hydraulic concentration [4]. At the operational scale, artisanal mineral processing further increases concentrations of dense radioactive minerals. Gravity concentration methods enrich high-density mineral fractions and therefore inevitably concentrate monazite, xenotime, and zircon alongside economically desirable minerals. Similar enrichment processes have been documented in heavy-mineral sand deposits and tantalum-mining operations elsewhere [40]. Consequently, elevated gamma dose rates measured in some concentrates should not be interpreted solely as a reflection of source-rock composition. Rather, they represent the cumulative effect of natural geological concentration and human-mediated beneficiation. Understanding this distinction matters because radiological significance may increase substantially as materials move through the mining and trading chain.

4.4. Implications for Illegal Mining, Mineral Supply Chains, and Radiological Screening

The results of this study demonstrate that materials traded as "coltan" or "black sands" within illegal mining networks of the northwestern Amazonian Craton are not only mineralogically heterogeneous but may also contain significant concentrations of naturally occurring radioactive materials (NORM). This finding extends beyond mineral resource evaluation because the extraction, transportation, storage, commercialization, and confiscation of these concentrates frequently occur without mineralogical characterization or radiological controls. Unlike industrial mining operations, informal and illegal mining activities generally lack routine mineralogical verification and radiation-monitoring procedures. Consequently, miners, transporters, traders, storage personnel, and law-enforcement agencies may unknowingly handle concentrates containing elevated concentrations of uranium- and thorium-bearing minerals (Supplementary Materials). The radiological significance of these materials is further complicated by the widespread commercial use of the term "coltan", which conceals substantial mineralogical variability among concentrates originating from different parts of the northwestern Amazonian Craton.

Radiometric measurements revealed a marked contrast between local background radiation and several confiscated mineral concentrates. Background radiation measured under field conditions averaged approximately 0.089 μSv/h, whereas laboratory background values ranged between 0.107 and 0.173 μSv/h (Supplementary Materials). In comparison, gamma dose rates in storage areas containing confiscated materials reached approximately 4 μSv/h, while directly above some monazite-rich concentrates they exceeded 20 μSv/h, and in samples provided by indigenous communities, even 70 μSv/h (Figure 7). These results indicate that certain concentrates generate radiation fields several orders of magnitude higher than local background conditions. Furthermore, measurements conducted at increasing distances from stored concentrates demonstrated rapid attenuation of gamma radiation, highlighting the importance of measurement geometry when evaluating potential exposure scenarios (Figure 3). The distinction between direct measurements performed above mineral concentrates and measurements obtained within storage facilities is particularly relevant, as these situations represent fundamentally different exposure conditions.

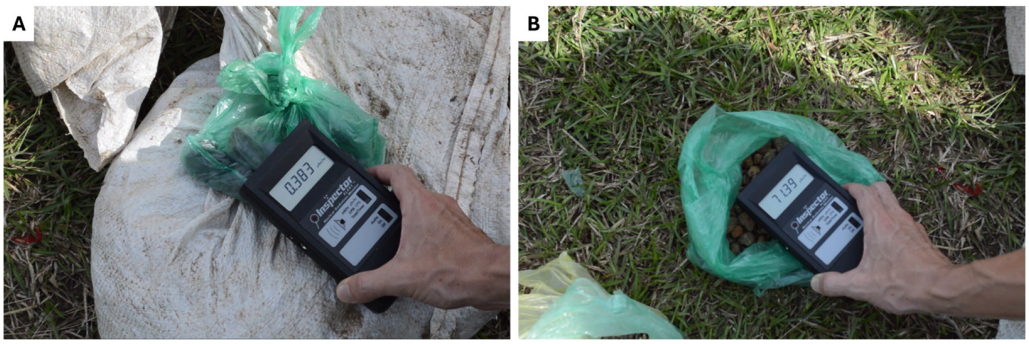


Figure 7. Gamma radiation measurements of mineral materials transported without protective measures. (**A**) Mineral material transported in a plastic bag by indigenous miners, showing low gamma (~0.4 μSv/h) radiation values measured directly at the material surface. (**B**) A second transported material exhibiting higher gamma radiation levels (71.4 μSv/h). In both cases, the materials are handled without personal protective equipment or radiological controls, illustrating variability in radioactivity among mineral concentrates and potential exposure risks to local communities.

The observed variability among samples also demonstrates that the radiological characteristics of confiscated materials cannot be reliably inferred from visual inspection or commercial classification alone. Materials transported under the same commercial designation may exhibit substantially different mineralogical compositions and radiological signatures

depending on the abundance of monazite, xenotime, zircon, and other NORM-bearing minerals. Consequently, routine mineralogical characterization and radiological screening could significantly improve management of confiscated materials, reduce uncertainty about their composition, and help identify concentrates that warrant further investigation.

The radiological results presented herein should nevertheless be interpreted as a screening assessment rather than a comprehensive risk evaluation. Measurements were obtained using a portable Geiger-Müller detector, which provides estimates of external gamma dose rates but does not identify radionuclides or quantify radionuclide-specific activity concentrations. Accordingly, the reported values should be regarded as indicators of radiological significance [8,9,24,26] rather than direct estimates of occupational exposure, effective dose, or environmental impact. More comprehensive assessments would require gamma spectrometry, radionuclide-specific activity determinations, airborne particulate monitoring, radon measurements, and personal dosimetry studies.

Despite these limitations, the measured dose rates demonstrate that certain confiscated concentrates generate radiation fields significantly above measured background conditions and therefore warrant further investigation. Similar associations between Nb-Ta mineralization, REE-bearing minerals, and NORM occurrence have been documented in heavy-mineral placer systems and tantalum-producing regions worldwide [7,37,38,40]. The coexistence of critical metals and radioactive minerals observed in this study therefore reflects a broader global relationship between critical-mineral resources and naturally occurring radioactivity. Within this context, the northwestern Amazonian Craton emerges as a region where critical-mineral exploration, illicit mineral trade, and NORM occurrence intersect. The results show that many materials marketed as "coltan" are in fact complex mixtures of Nb-Ta-bearing, REE-bearing, and radioactive heavy minerals. Improved mineralogical characterization and routine radiological screening would therefore benefit not only resource evaluation and regulatory oversight but also miners, traders, local communities, and law-enforcement agencies that may come into contact with these materials during extraction, transportation, storage, commercialization, and confiscation. More broadly, these findings highlight the need to move beyond generic commercial terminology and adopt analytical approaches that can accurately identify the mineralogical and radiological characteristics of concentrates circulating through regional and international supply chains.

5. Conclusions

Materials marketed as "coltan" within illegal mineral supply chains of the northwestern Amazonian Craton do not correspond exclusively to columbite–tantalite but instead comprise heterogeneous assemblages of Nb-Ta-bearing, REE-bearing, and NORM-bearing heavy minerals, including rutile-group minerals, wolframite, monazite, xenotime, zircon, ilmenite, and iron oxides.

Integrated XRD and WDXRF analyses indicate that Nb and Ta are predominantly associated with rutile-group minerals, particularly ilmenorutile and strüverite, whereas REEs, uranium, and thorium occur mainly within monazite–xenotime assemblages. These results demonstrate that the mineralogical diversity of materials traded as "coltan" extends well beyond the traditional columbite–tantalite definition.

Elevated gamma dose rates measured in monazite-bearing concentrates are consistent with enrichment of naturally occurring radioactive materials (NORM) through natural placer-forming processes and subsequent artisanal mineral concentration. Although this study represents a radiological screening assessment rather than a comprehensive risk evaluation, the measured dose rates highlight the radiological significance of some materials circulating through illegal mining and trading networks.

The widespread misclassification of these concentrates highlights the need for routine mineralogical characterization and radiological screening to support resource evaluation, improve traceability and regulatory oversight, and strengthen management strategies for miners, traders, local communities, and law-enforcement agencies involved in extracting, transporting, storing, commercializing, and confiscating these materials. The marked mineralogical variability observed among confiscated samples further suggests that concentrates marketed as "coltan" or "black sands" may include mixtures of materials derived from different source areas within the northwestern Amazonian Craton. Such mixing may inadvertently or intentionally obscure the presence of higher-value REE-rich and NORM-bearing concentrates within illegal supply chains, complicating resource governance, traceability, and radiological control.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/earth7050164/s1, File S1: Supplemantary_Data_DRX; Table S1: Supplementary_Data_Samples; Table S2: Supplementary_Data_Geochemistry; Table S3: Supplementary_Data_Radiactivity; Video S1: Supplementary Data radioactivity.

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