The joint virtual event of the
African Light Source AfLS-2025 (8th)
and the
African Physical Society AfPS2025

Discrimination of Colombo-tantalite minerals from South Kivu Province (DRC) using XRF techniques

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Outline

1. Introduction / Context
2. Materials and Methods
3. Results and discussion
4. Conclusion/Recommendations

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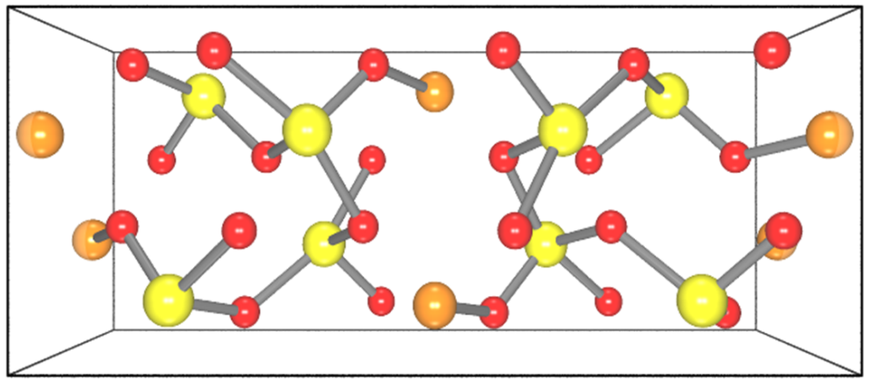
Coltan = Colombite-tantalite or colombo-tantalite

$Fe(Ta,Nb)_2O_6$ Structure

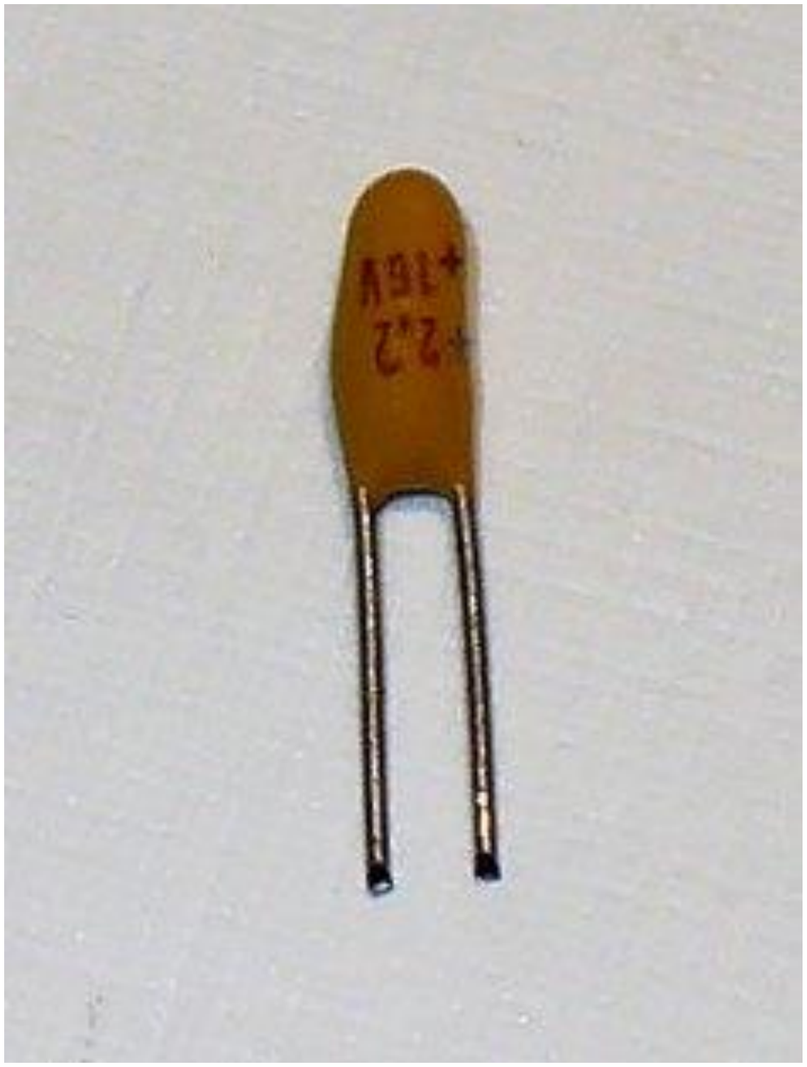


- Red balls: oxygen, orange balls: Fe and Mn (for coltan), yellow balls: Ta and Nb,
- Orthorhombic structure with **Pbcn** space group[2]
- Coordination: 2 for the Fe atoms and 5 for Ta,Nb,
- Paramagnetic,
- Density 5.20 – 8.20 g/cm³.

Nb and Ta properties

Property	Ta	Nb	Comments
Abundance in Earth continental crust (ppm)	0.7	8	Rare metals
Mohs hardness (MPa)	6.6	6.0	diamond: 10, Ti: 6
Melting point (°C)	2996	2468	Top 6 among metals
Density (g/cm³)	16.65	8.57	
Superconducting Tc	4.47	9.25	Nb has the highest Tc among the elements
Reactivity	impervious to chemical attacks	reacts with halogens, C, O_2, N_2, S	

Utilisation of Ta



- Ta electrode/Ta_2O_5 dielectric interface: capacitor for energy storage (Apple, Samsung, etc.)
- thermal resistance and corrosion prevention,
- TaC coating (strength layers)
- etc.

Utilisation of Nb

- Orthopaedic implants
- steel, super alloys for aerospace equipment, superconducting coils (Nb-Ti alloy), etc.

Millau Valley bridge (FR)
0.025% niobium => 60% weight reduction

Gibson, 2016

Moisés Gómez, Jinhui Li, Xianlai Zeng, (2024) Resources, Conservation and Recycling, **209**, 107744

Øresund Bridge (*Sweden-Denmark*)
0.022% niobium
=> weight reduction 15 kt
=> saved money : US$25 million

Bakry et al., 2023

https://niobium.tech/en

Coltan = Colombite-tantalite
Conflict minerals => 3TG

Cassiterite: Sn

Coltan: Ta, Nb

Wolframite: W

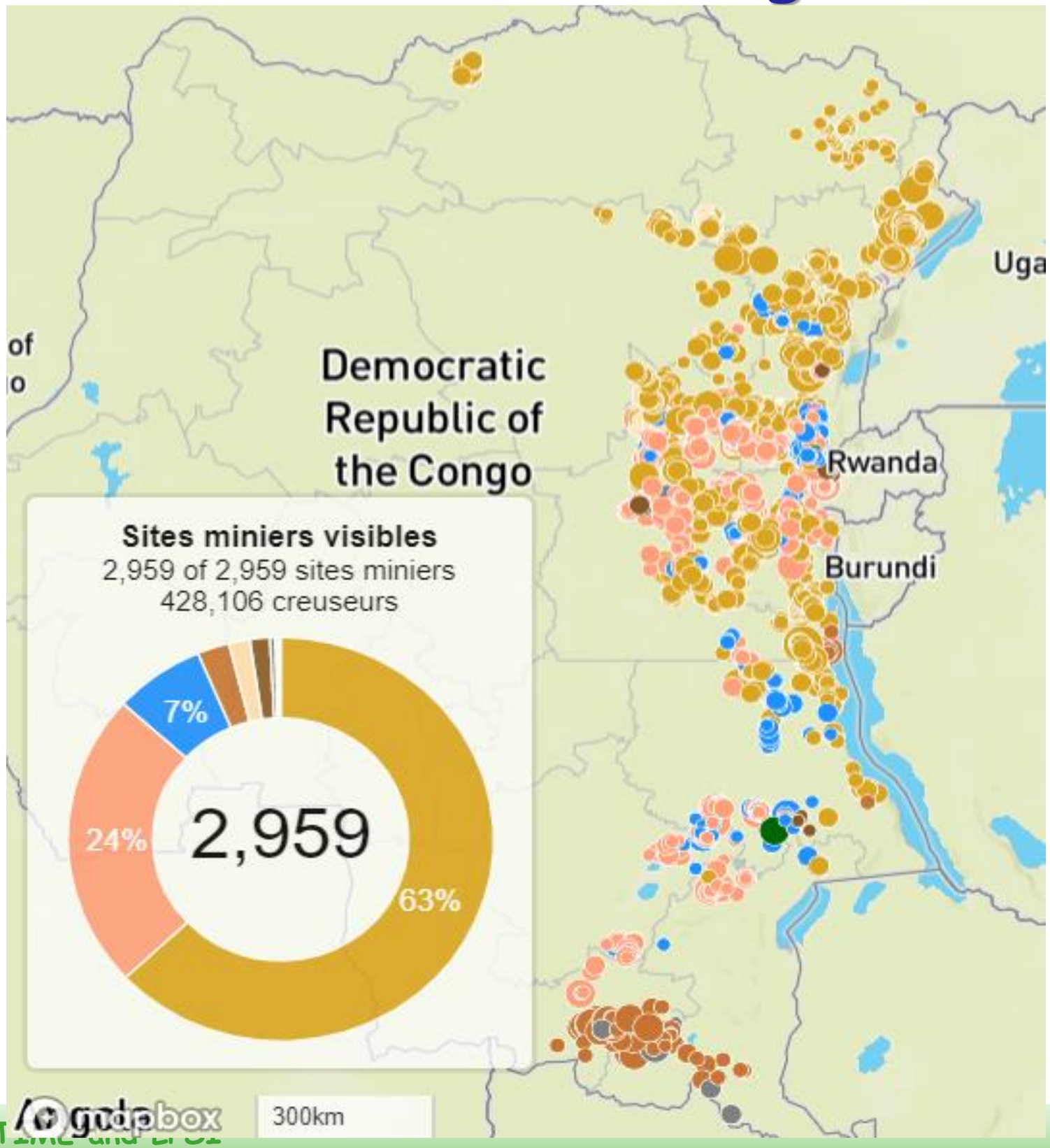
Gold: Au

http://www.l-integration.com/?p=6797

J. Hayes and R. Burge, 2008, Fauna and Flora Int. 2003

http://www.consoglobe.com/coltan-metal-sanglant-dans-telephones-cg

Artisanal Mining Sites in DRC



Carte de l'exploitation minière artisanale dans l'Est de la RD Congo — IPIS

Année de visite

2009 2010 2011 2012 2013 2014 2015 2016 2017 2018 2019 2020

Jeux de données

- **Sites miniers artisanaux**
  - Substances minérales
    - Or
    - Cassitérite
    - Coltan
    - Wolframite
    - Diamant
    - More ...
  - Présence armée
    - Pas de présence armée constatée
    - FARDC - Pas de données sur les ingérences
    - FARDC - Pas d'ingérence constatée
    - FARDC - Éléments indisciplinés
    - Groupe armé local
    - Groupe armé étranger
    - PNC - Éléments indisciplinés

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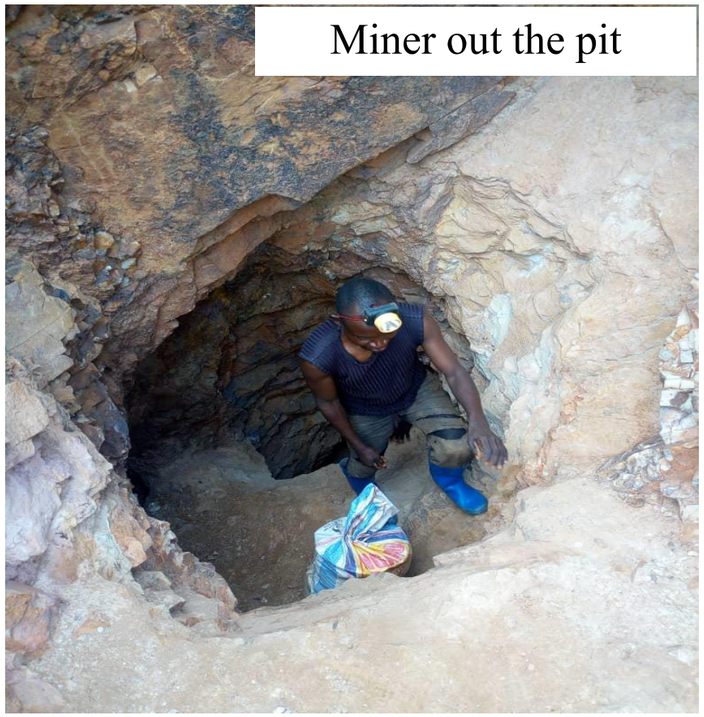
Artisanal mining and processing of 3TG

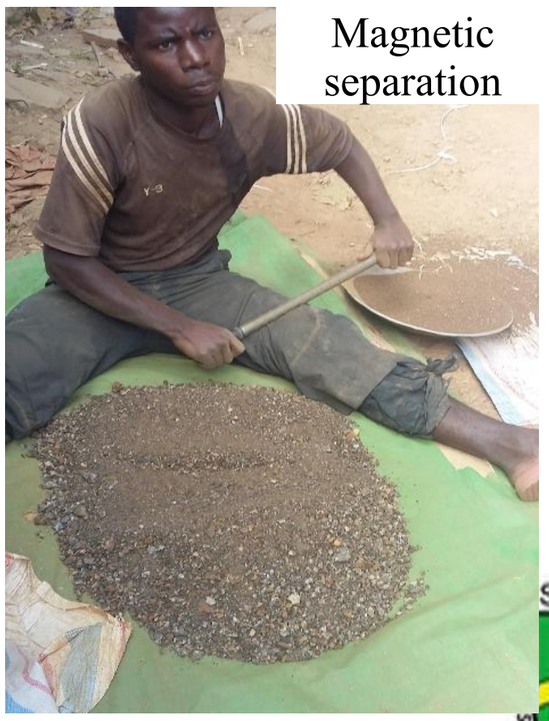
Miners at new unsecured pit

Miners cleaning cassiterite in the river



Miner out the pit

Ore separation in the river



Magnetic separation

Aware on poor daily work conditions (on site visit 2018)

Kivu's coltan: A facility for chemical-physics treatment and application study

Four work packages since 2019

1. Sustainable techniques for purification and separation of Ta and Nb are locally available.
2. New channels to extended Ta and Nb valorization are identified while strengthening local expertise.
3. Minerals from the certified sites are better characterized to improve information to operators /artisanal miners on the value, precautions/safety or traceability.
4. Recommendations for the better interventions and the improvement of the artisanal mining sector are formulated.

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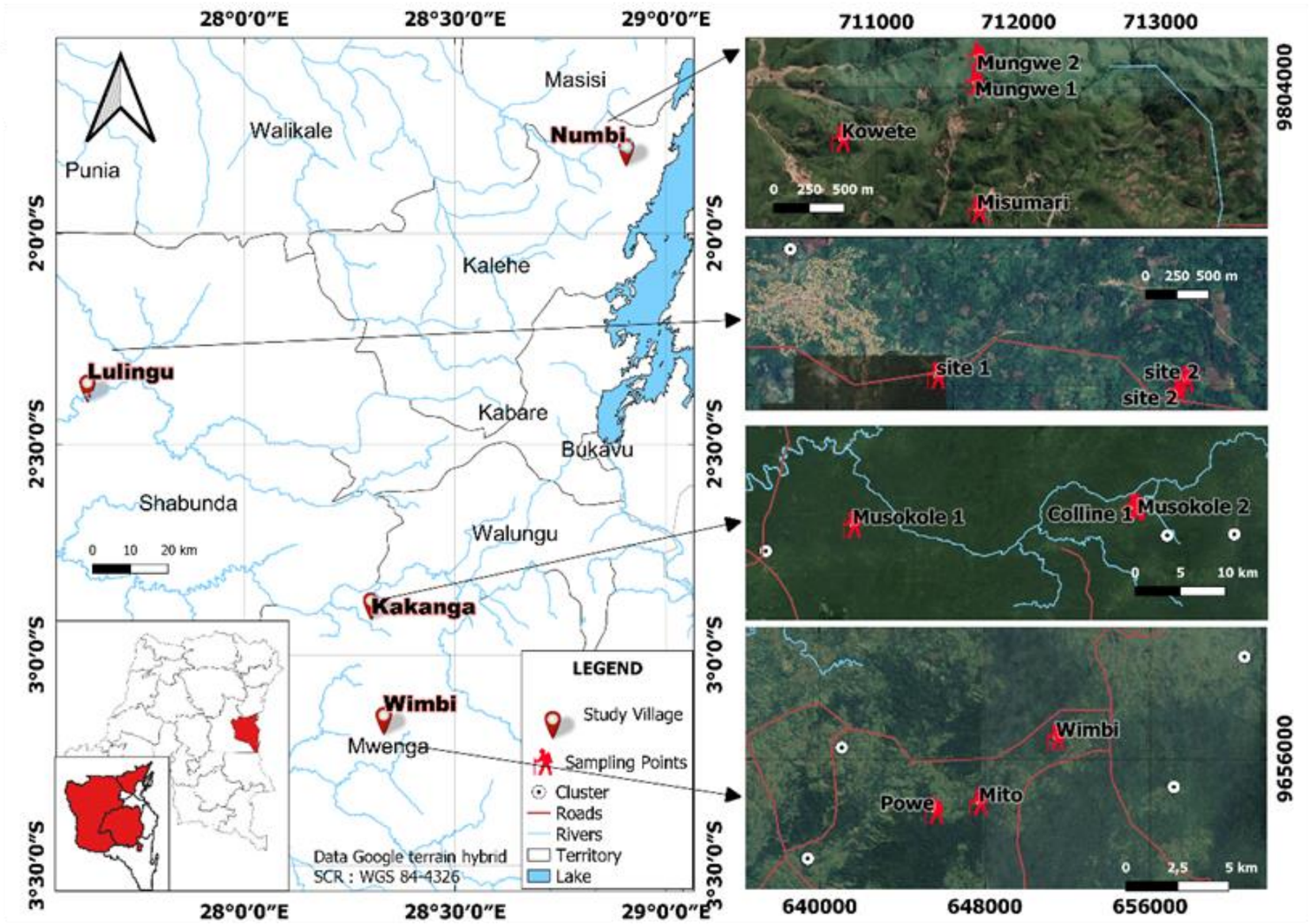
Materials et methods: sampling

(a)



(b)

(c)

Samples are collected from certified sites according to iTSCi standards [1]: the so called green flagged sites.

Radex portable Geiger-Müller

effective dose (expressed in µSv/h),

10 kg per site

Oven drying up to 90°C

RM200 grinder < 45 micrometer

1-https://www.itsci.org

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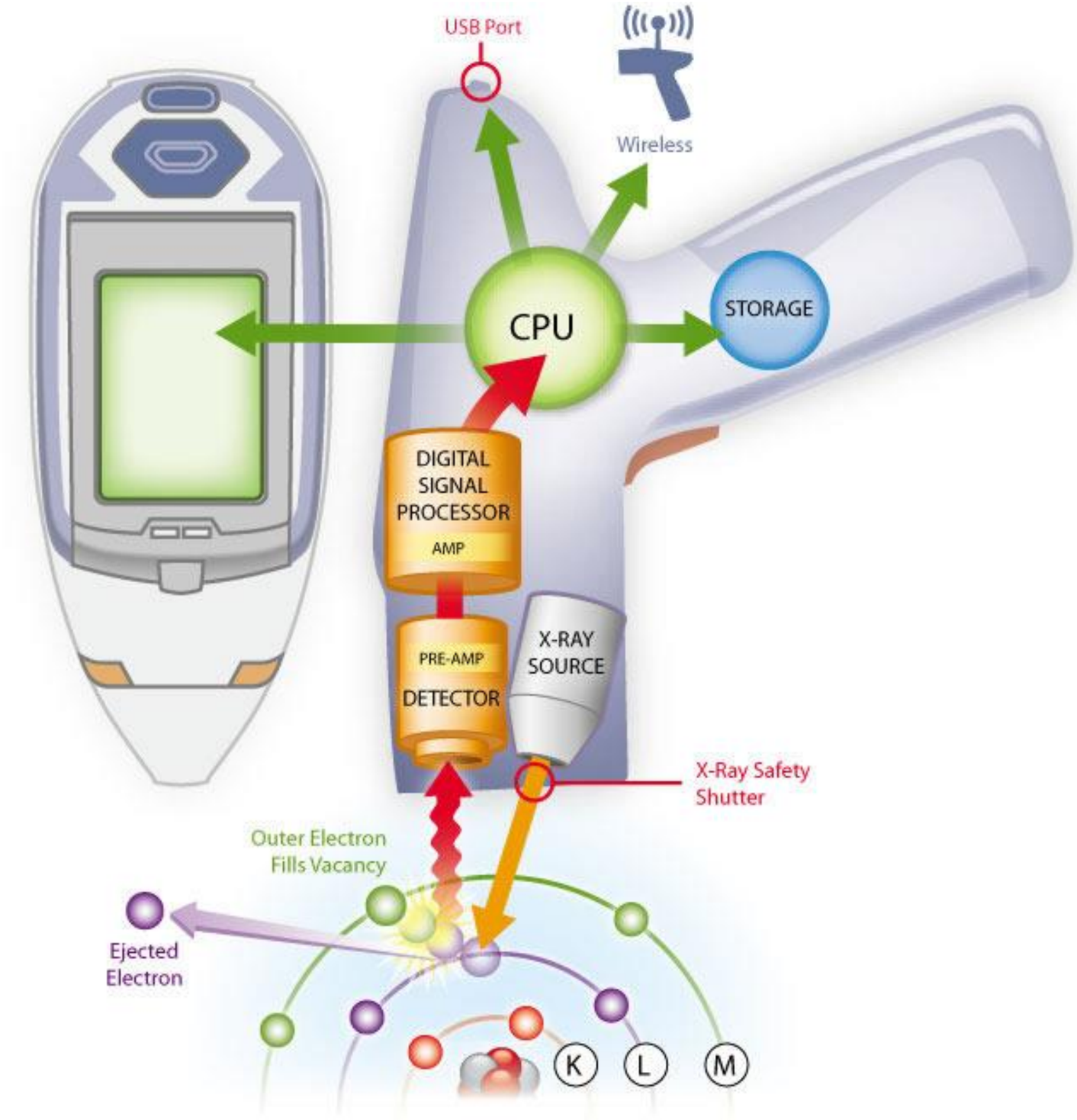
Materials et methods: characterisation



Chemical analysis

- Local characterization of samples by X-ray fluorescence analysis (XRF, weight: 1.9 kg, powered by a Li-ion battery) using a portable equipment before and after purification process. Quantitative analysis of the elements from Mg to U.

- Complementary analysis by proton induced x-ray emission (PIXE), X-ray photoemission (XPS), etc. are planed

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Materials et methods: characterisation

Mineralogical inspection

- Two different diffractometers were used to identify the minerals bearing the studied samples.
- X-ray diffraction analysis of the Numbi and Lulingu samples was performed using a Bruker D8-ECO diffractometer,
- while that of Kakanga and Wimbi was performed using a Panalytical X'Pert PRO.
- X-ray diffraction analysis was performed with CuKα radiations (λ = 1.54 Å).
- For this purpose, the sample was scanned between 3° and 70°2 θ (or 90°2θ) at a rate of 0.02°2θ per second.
- The identification of all phases from the XRD patterns was carried out using a Panalytical Xpert suite and WebPDF4 + ICDD relational database, and completed with Match software.

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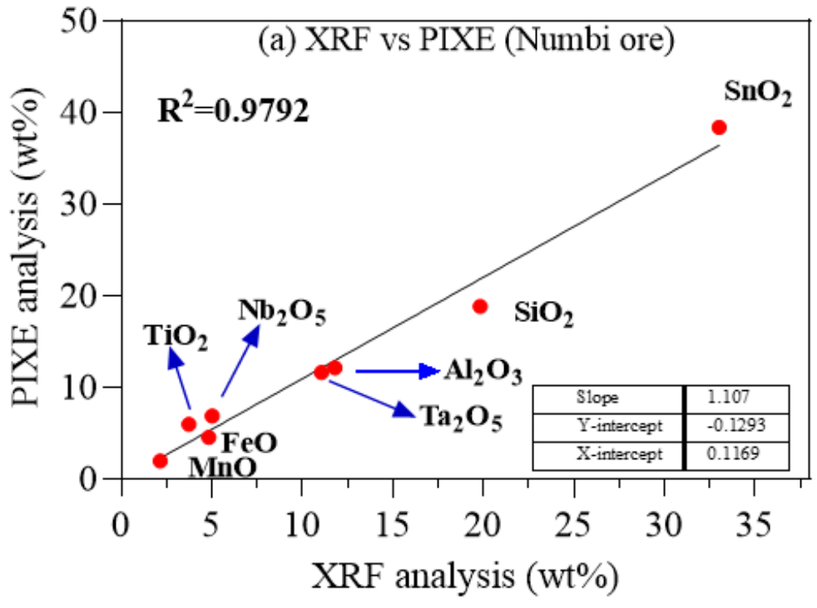
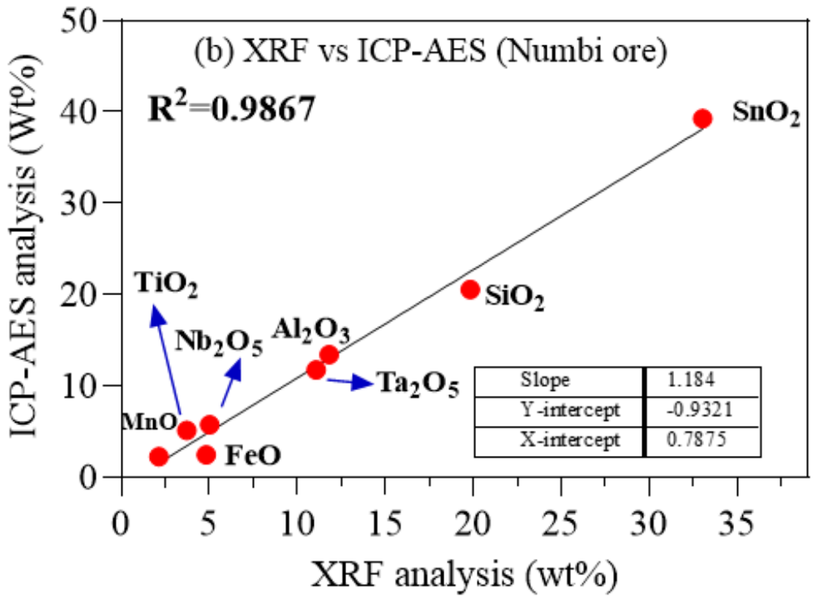
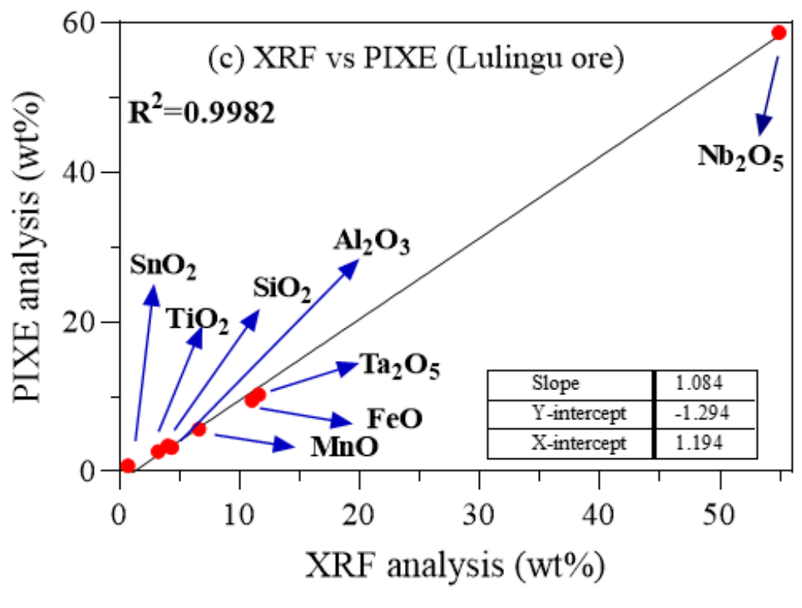
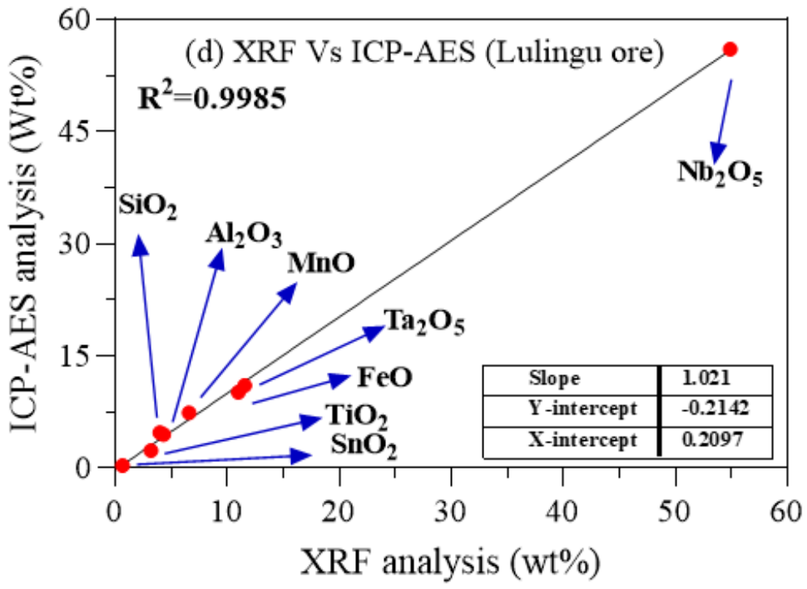
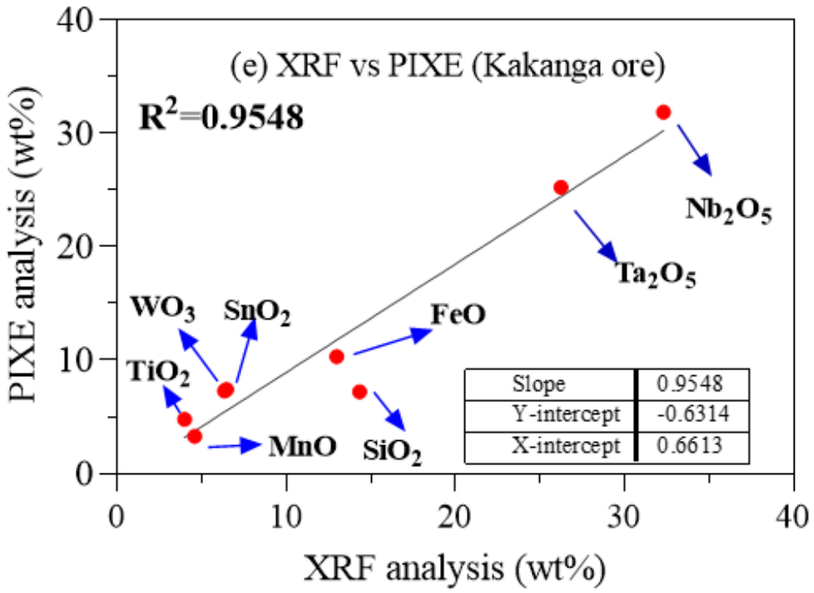
Results: Correlation between XRF, PIXE and ICP-AES

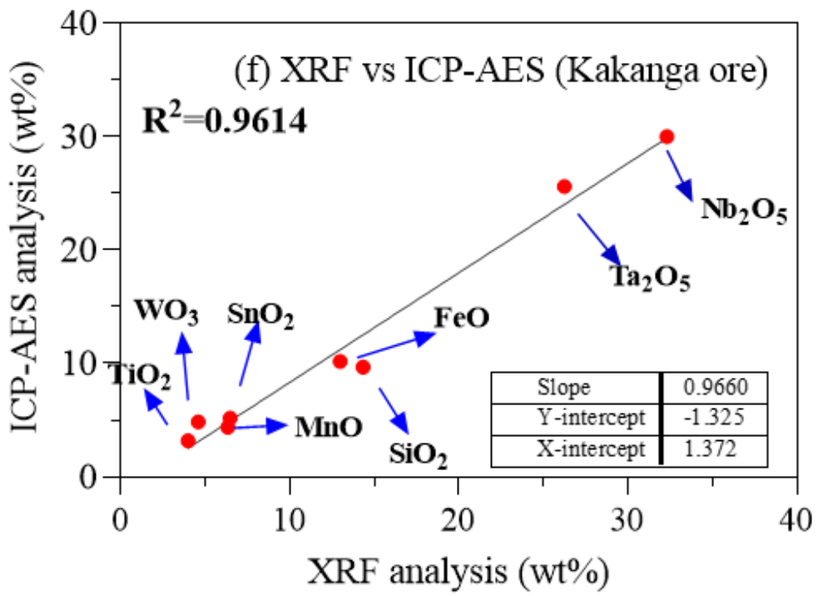






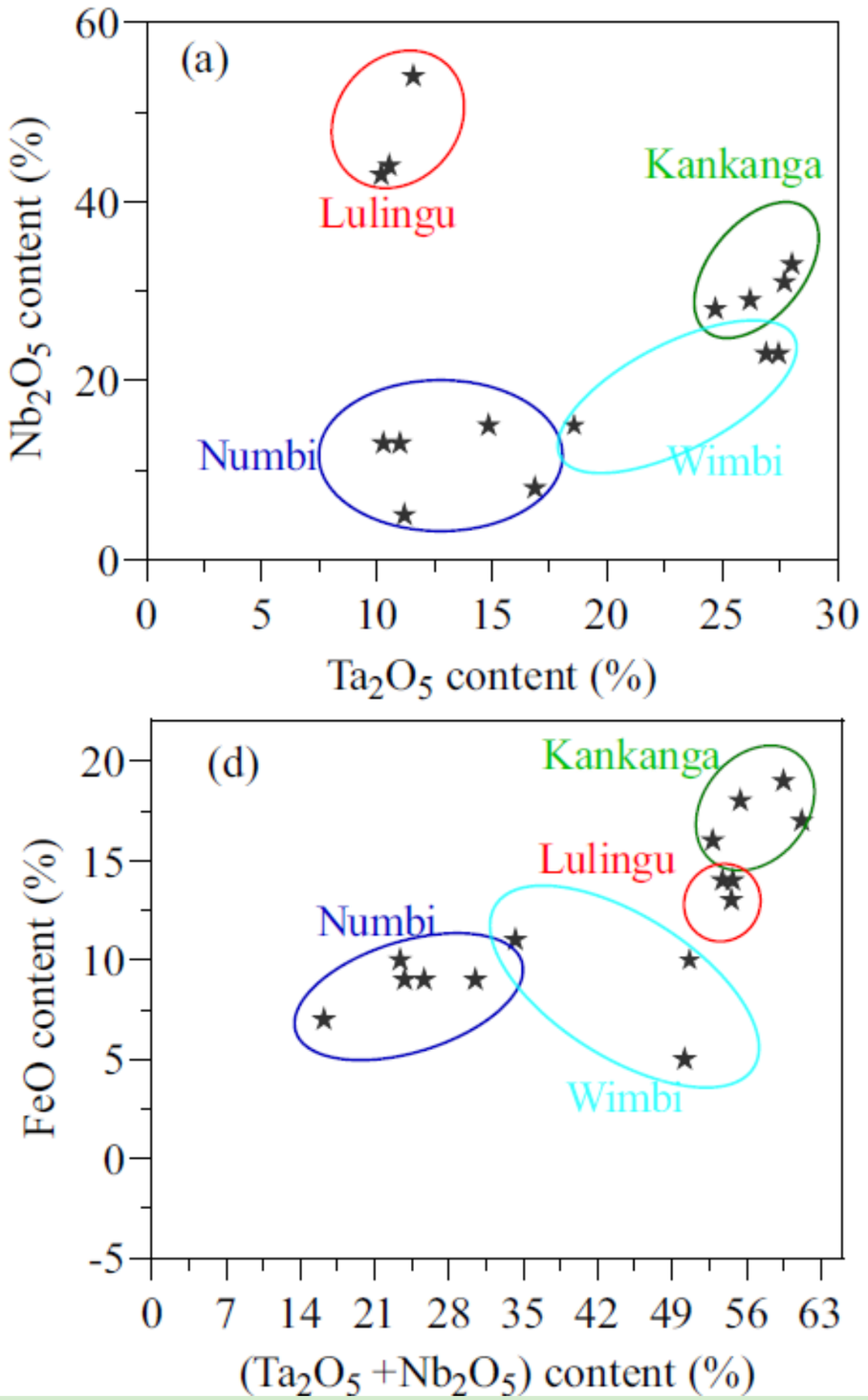


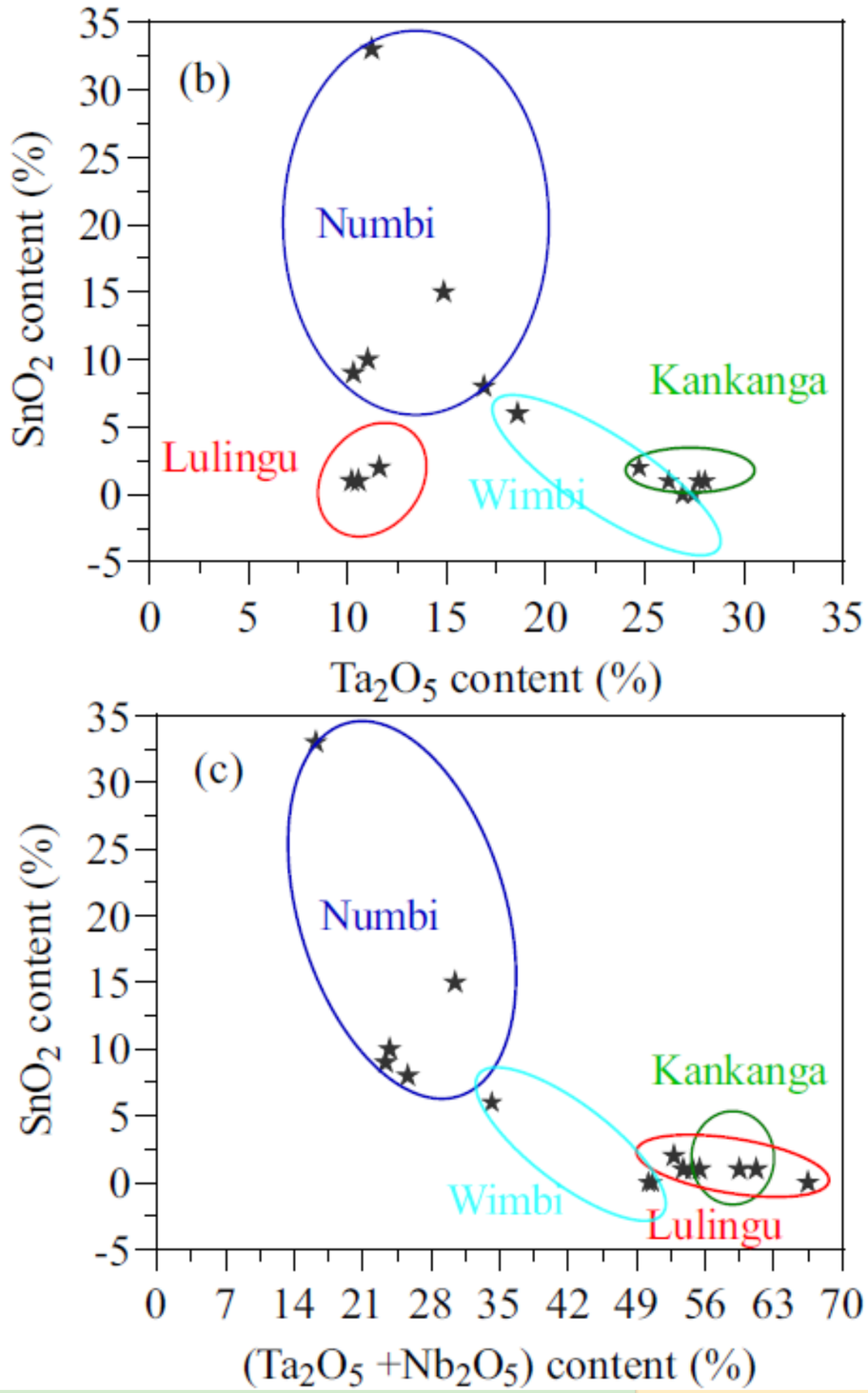




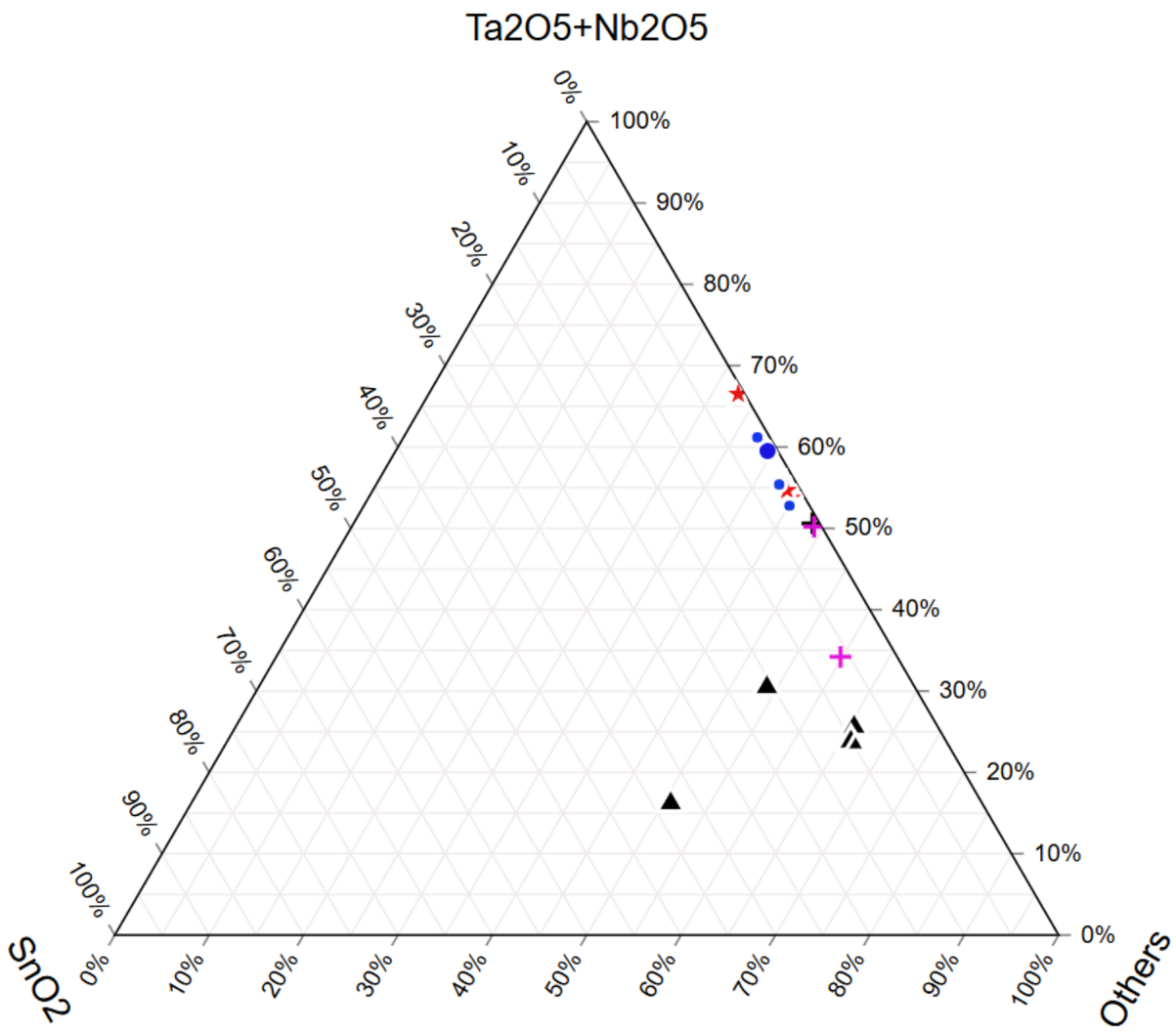
Very good correlation for the main elements (Ta, Nb, Fe, Mn, Sn, Mn) for three samples

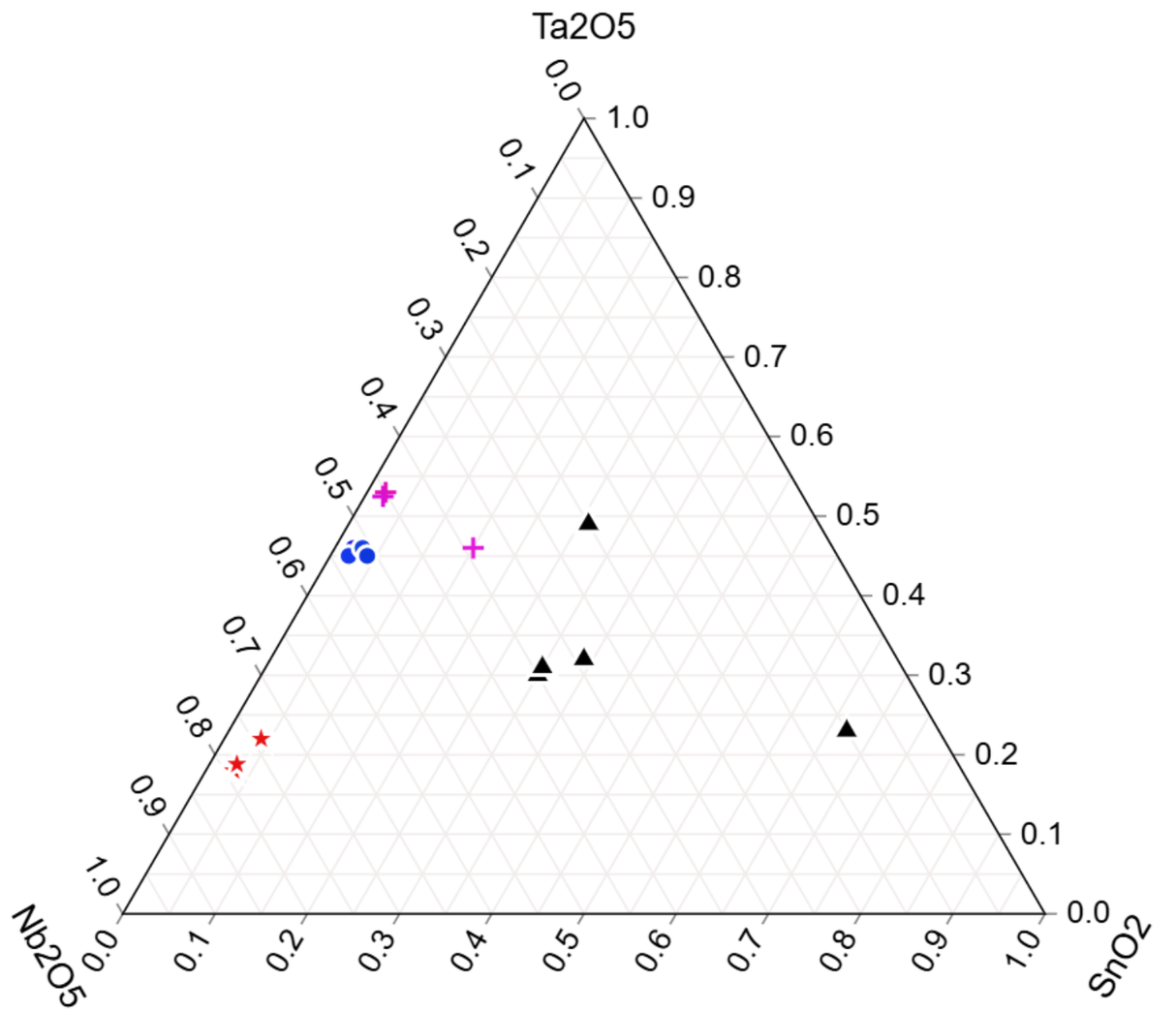
Results: Nb vs Ta, Sn vs Ta, Sn vs (Ta+Nb) and Fe vs (Ta+Nb).





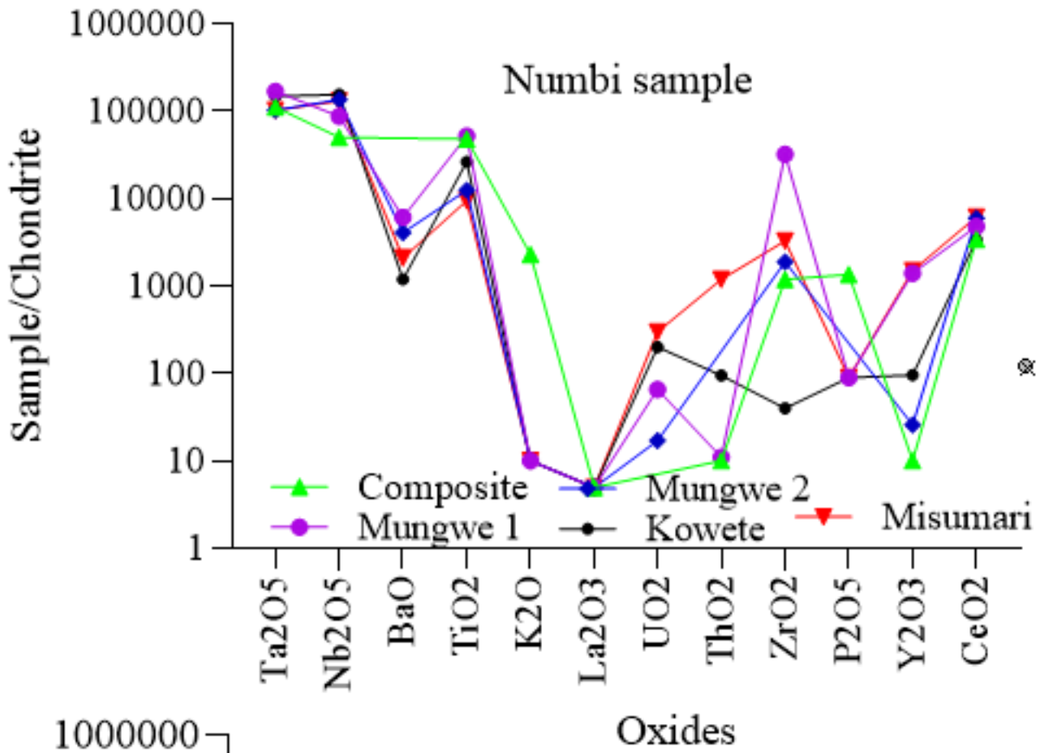
Results: ternary diagram

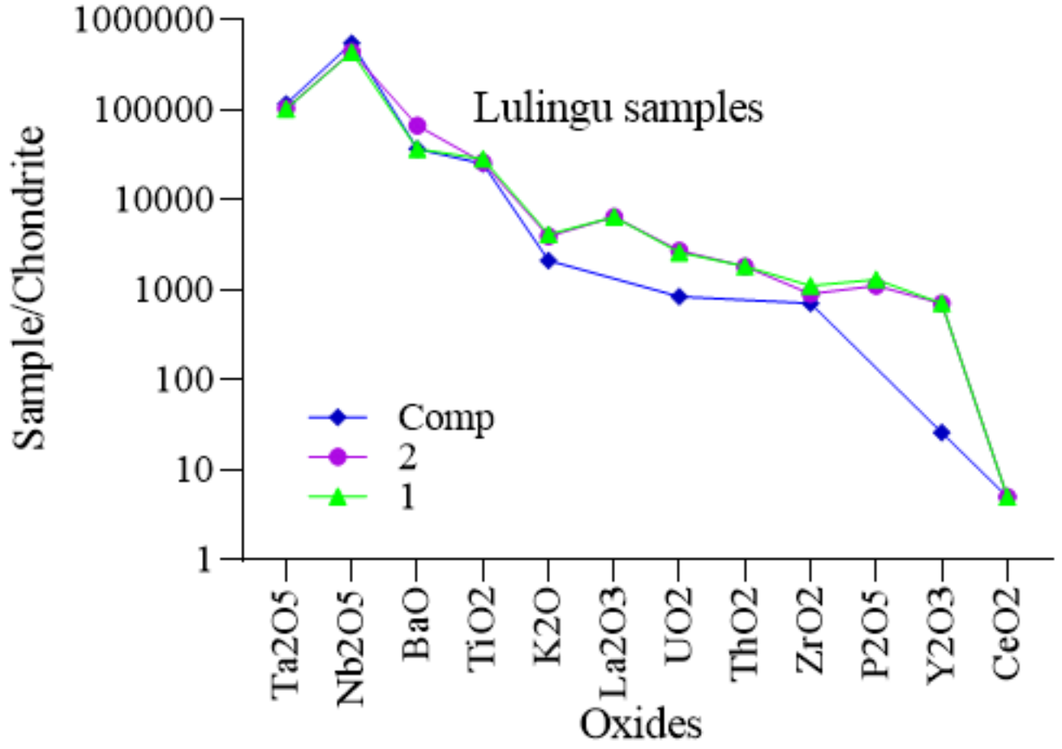


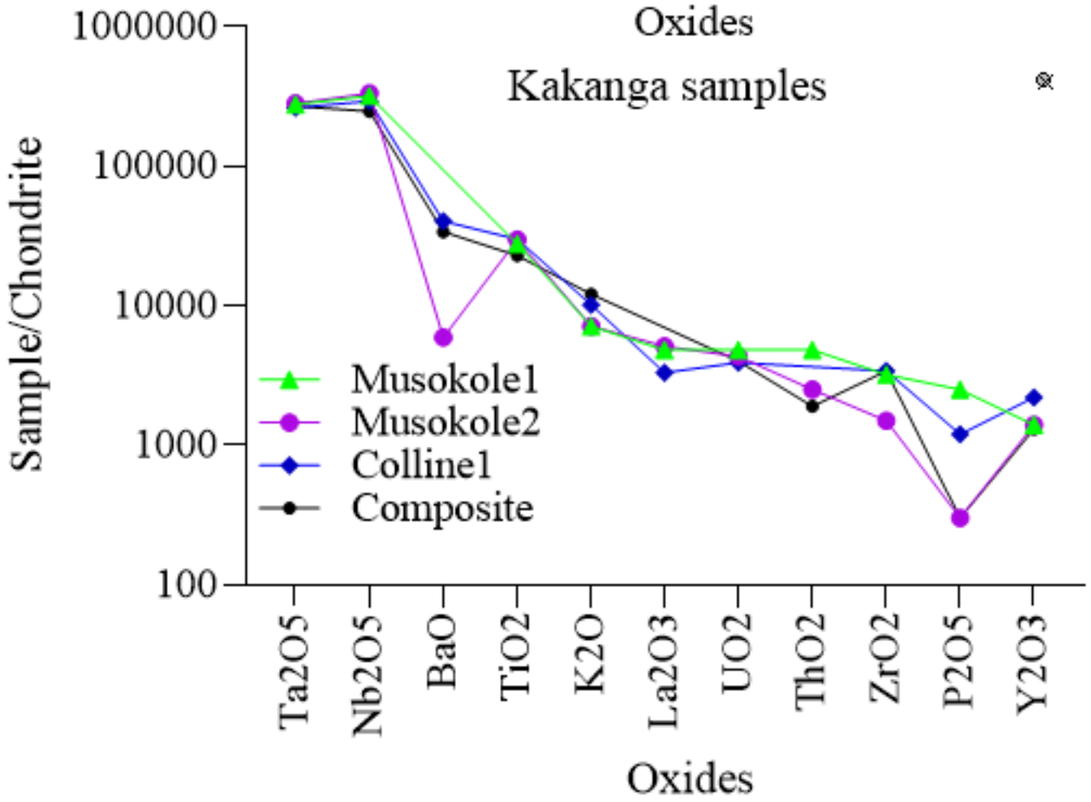


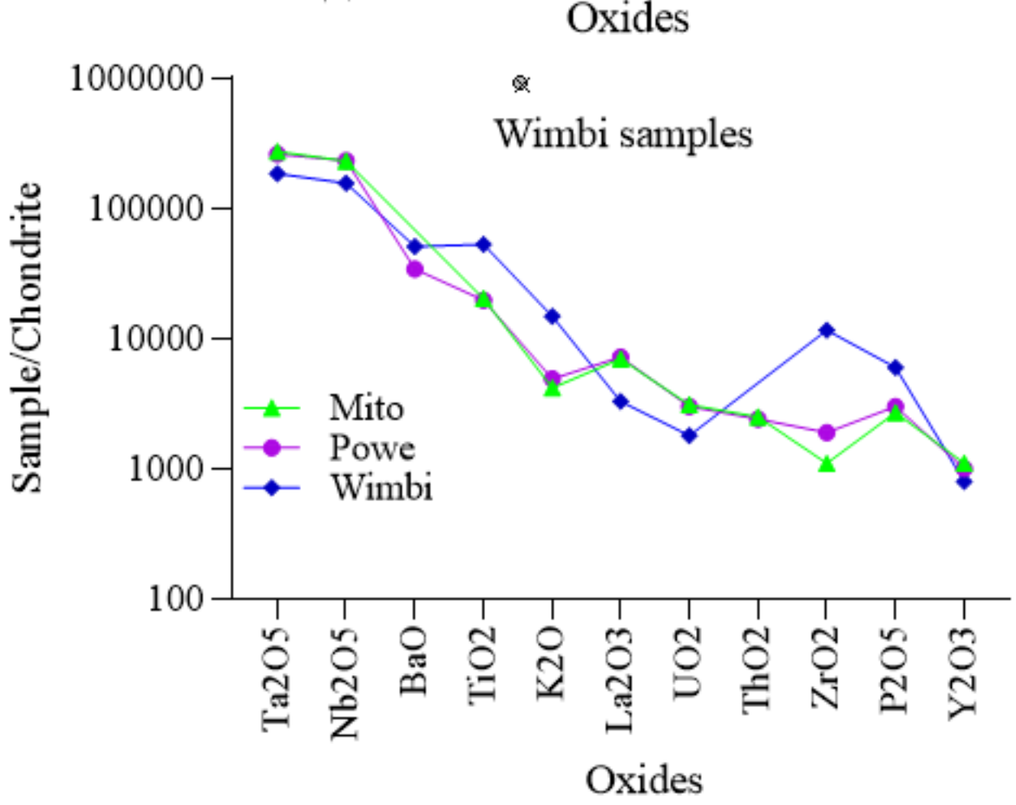
Discrimination of the columbite-tantalite ore samples based on ternary diagrams of the bulk compositon (a) the Sn-(Ta+Nb)-Others (b) Nb-Ta-Sn (▲, ★, ●, + symbols represent the samples from Numbi, Lulingu, Kakanga and Wimbi, respectively).

Results: spider diagram



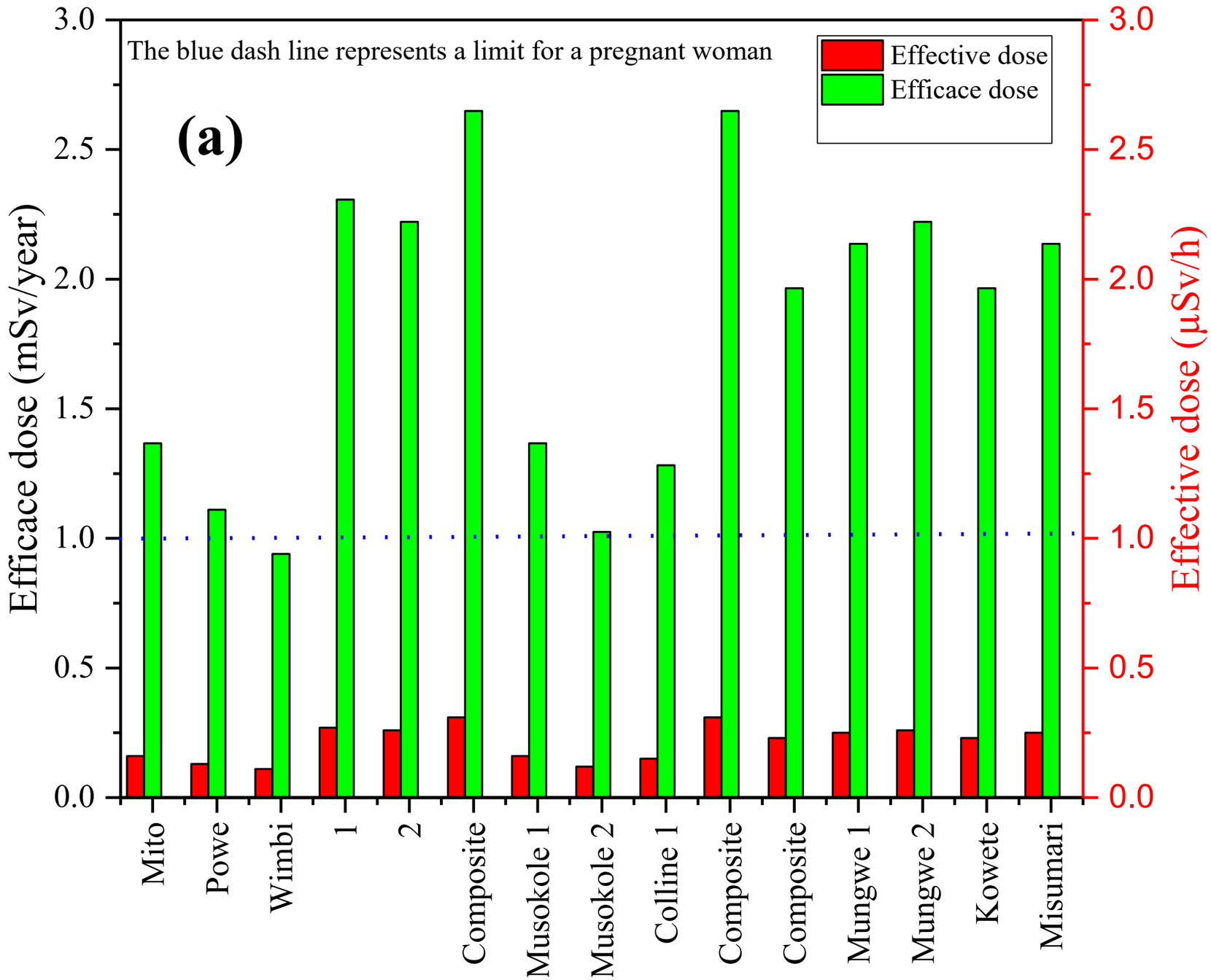






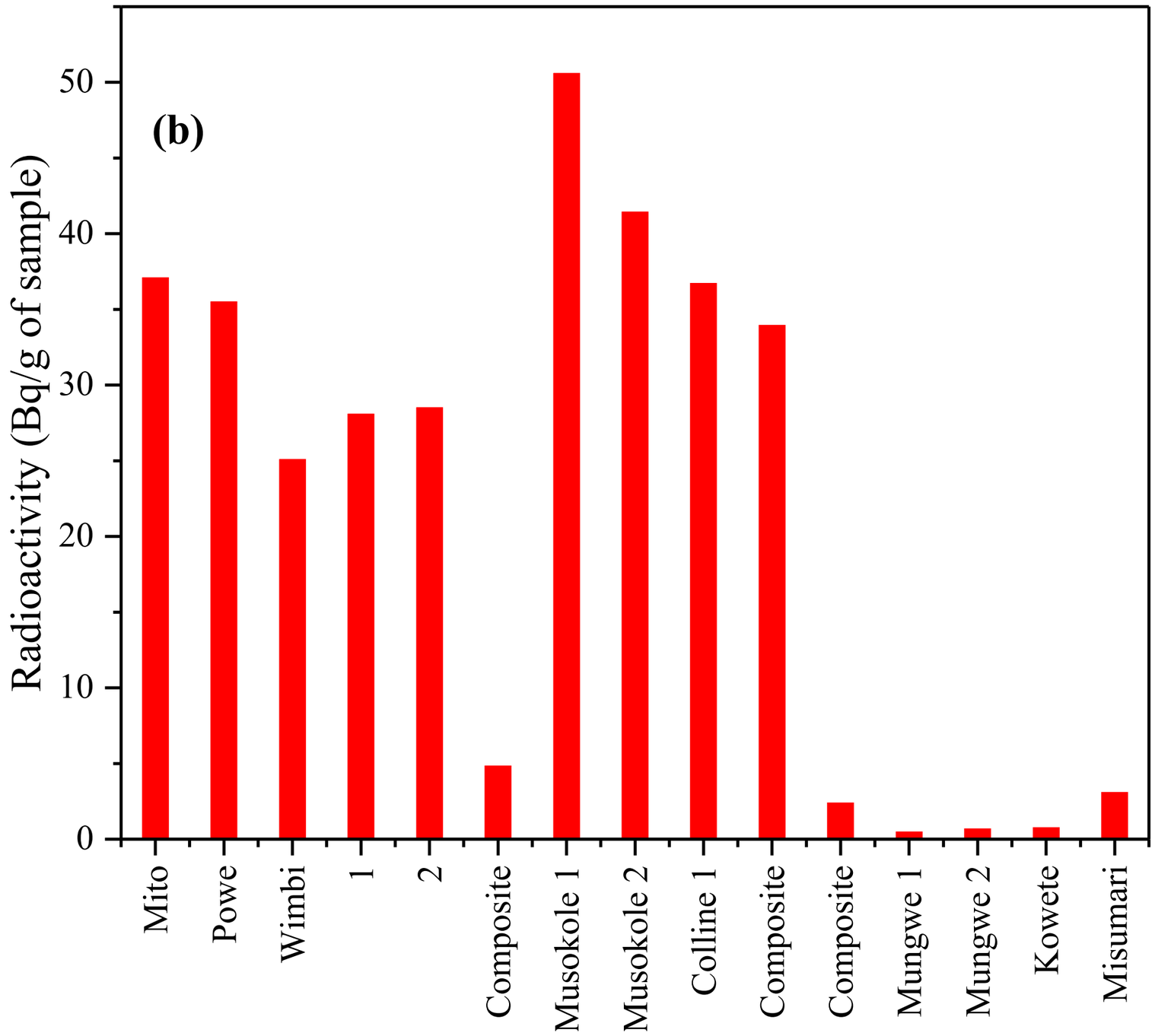
Samples of the same origin show an almost unique spider diagram pattern

Results: Effective dose



Effective doses are above the effective dose limit for pregnant women in 14 of the 15 studied samples

Results: Radioctivity



Radioactivity of the ore samples and composites: except the samples from Numbi, the other samples have a relatively high level of radioactivity. Materials below 10 Bq/g are exempt from the regulations for the transport of radioactive materials (Class 7)

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Conclusion / recommendations

- ✓ Portable XRF spectrometer as tool to fingerprint columbite-tantalite ores,
- ✓ XRF data compared with PIXE and ICP-AES and very good correlations were obtained ($R^2 > 0.95$).
- ✓ Mineralogical analyses and the results showed that columbite-tantalite appears in the form of a columbite phase, which is a combination of Nb, Ta, Fe and Mn.
- ✓ Discrimination diagrams were displayed and all show that columbite-tantalite ores can be discriminated from one to another according to their origins.
- ✓ Samples contain Ta and Nb, as well as important metals such as titanium and tin: to consider in the price of Congolese raw materials.
- ✓ Chemical analyses also revealed the presence of radioactive metals, which calls for recommendations for artisanal miners when exposed to these materials.
- ✓ The doses appear to be low in some cases. Pregnant women are most affected and their presence in the mines should be discouraged.
- ✓ Serious environmental damage ! No information on the radiation dose from the radioactive dust inhaled during columbite-tantalite extraction and processing. The initial radioactivity is almost entirely contained in the ore residue and is therefore seriously contaminating the region's waterways.
- ✓ Hopefully, our conclusions will provide decision-makers, artisanal miners, researchers, and investors with elements that will enhance their knowledge of columbite-tantalite ore fingerprints in the DRC

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Acknowledgements

A. Shikika, N. Aokowa, S. Ngayirwa, A. Mushamalirwa, G Ameli, I. Maluzi, C. Muntu wa Mwami, F. Zabene, F.A. Muvundja

A significant part of data used was part of the PhD Thesis of Dr. Ing. Alidor Shikika, which has been supported by the ARES-CCD 2019-2024 (Belgium).

Another part of the data set belongs to the Master Thesis of Nathan Aokowa and Sophie Ngayirwa.

SEM-EDS, ICP-OES, and additional XRD analysis were done at the GeMMe of the University of Liège.

PIXE and XRD data were done at the University of Namur.

To better appreciate our achievements, please read:

- ✓ Alidor Shikika, M.C. Mugumaoderha et al, Characterization and Geochemical Fingerprinting of Columbite-Tantalite Minerals From South Kivu Province (DRC) Using Portable XRF Techniques, Resource Geology 75 (1), e70014, 2025. https://doi.org/10.1111/rge.70014
- ✓ A. Shikika, M.C. Mugumaoderha et al, A review on extractive metallurgy of tantalum and niobium, Hydrometallurgy 198, 105496, 2020.
- ✓ A. Shikika, M.C. Mugumaoderha et al, Extraction of Nb and Ta from a coltan ore from South Kivu in the DRC by alkaline roasting – thermodynamic and kinetic aspects, Mineral Engineering 163, 106751, 2021.
- ✓ A. Shikika, M.C. Mugumaoderha et al, Extraction of Ta and Nb from a coltan Bearing Ore by mean of Ammonium Bifluoride Fluorination and Sulfuric Acid Leaching, Minerals 11, 1392, 2021. https://doi.org/10.3390/min11121392
- ✓ A. Shikika, M.C. Mugumaoderha et al, Efficient Extraction of Ta and Nb from a Coltan Ore Through Alkaline Roasting, Water Leaching, Precipitation, and Oxalic Acid Leaching, Journal of Sustainable Metallurgy 8, 1932, 2022. https://doi.org/10.1007/s40831-022-00621-w
- ✓ A. Shikika, F. Muvundja, M. C. Mugumaoderha and St. Gaydardzhiev, Separation and purification of Ta and Nb from ammonium bifluoride leachates using methyl isobuthyl ketone, 2-Octanol and Aliquat® 336, Journal of Sustainable Metallurgy, 2024