TU Delft

Delft University of Technology

Document Version
Final published version

Citation (APA)
Guatame-Garcia, A., Buxton, M., Tinti, F., Kasmaeeyazdi, S., Bodenan, F., & Schick, J. (2023). Residue sampling and characterization. In O. Chernoburova, & A. Chagnes (Eds.), *Mining and Processing Residues: Future's Source of Critical Raw Materials* (pp. 25-69). Elsevier. https://doi.org/10.1016/B978-0-323-95175-3.00002-7

Important note
To cite this publication, please use the final published version (if applicable).
Please check the document version above.

Copyright
In case the licence states “Dutch Copyright Act (Article 25fa)”, this publication was made available Green Open Access via the TU Delft Institutional Repository pursuant to Dutch Copyright Act (Article 25fa, the Taverne amendment). This provision does not affect copyright ownership.
Unless copyright is transferred by contract or statute, it remains with the copyright holder.

Sharing and reuse
Other than for strictly personal use, it is not permitted to download, forward or distribute the text or part of it, without the consent of the author(s) and/or copyright holder(s), unless the work is under an open content license such as Creative Commons.

Takedown policy
Please contact us and provide details if you believe this document breaches copyrights.
We will remove access to the work immediately and investigate your claim.

This work is downloaded from Delft University of Technology.

Green Open Access added to TU Delft Institutional Repository

'You share, we take care!' - Taverne project

https://www.openaccess.nl/en/you-share-we-take-care

Otherwise as indicated in the copyright section: the publisher is the copyright holder of this work and the author uses the Dutch legislation to make this work public.

2

Residue sampling and characterization

Adriana Guatame-Garcia[1,2], Mike Buxton[1], Francesco Tinti[3], Sara Kasmaeeyazdi[3], Francoise Bodenan[4], Joachim Schick[5]

[1]DEPARTMENT OF GEOSCIENCES AND ENGINEERING, DELFT UNIVERSITY OF TECHNOLOGY, DELFT, THE NETHERLANDS [2]DEPARTMENT OF GEOLOGICAL SCIENCES & GEOLOGICAL ENGINEERING, QUEEN'S UNIVERSITY, KINGSTON, ON, CANADA [3]DEPARTMENT OF CIVIL, CHEMICAL, ENVIRONMENTAL AND MATERIALS ENGINEERING, UNIVERSITY OF BOLOGNA, BOLOGNA, ITALY [4]BRGM (BUREAU DE RECHERCHES GÉOLOGIQUES ET MINIÈRES), ORLÉANS, FRANCE [5]INNOVATION CENTER FOR EXTRACTIVE METALLURGY, ORANO MINING, BESSINES-SUR-GARTEMPE, FRANCE

2.1 Introduction

The idea of sourcing critical raw materials (CRMs) from mine tailings and waste rocks surges from (1) the need for contributing to the supply of metals that are critical for the economy and society, (2) the economic stimuli to support the already costly environmental management, and (3) the improvements in extraction and processing efficiency of mineral resources in general. For achieving resource efficiency, it is necessary to implement improved mining and processing methods. Buxton and Benndorf (2013) stated that material characterization using sensor-derived data enables the optimization of the processes along the mining value chain. It does so by, among others, discriminating ore from waste, ensuring the quality and homogeneity of the material that is fed at different stages of mining and processing, and ensuring the product's adherence to quality specifications.

It is then understood that to ensure the success in the recovery of CRMs from mine waste deposits, it is necessary to elucidate the attributes not only of the ore (CRMs in this particular case) but also of the host material in general. For doing this, it is essential first to define the scope and stages of a project to explore mine residues as a possible resource (Dominy, 2018). Then, decisions must be made depending on the type of material that needs to be analyzed and the best analytical techniques to identify all the relevant attributes. In this sense, a comprehensive understanding of mine waste material targeting CRMs potential is facilitated by implementing a sensor-based approach for material characterization.

The characterization of mine residues in the view of mineral recovery must provide the following information:

- the occurrence of recoverable minerals and their sources;
- the possible distribution of recoverable minerals inside the waste deposit;

Mining and Processing Residues. DOI: https://doi.org/10.1016/B978-0-323-95175-3.00002-7
© 2023 Elsevier Inc. All rights reserved.

- the mechanisms of metal enrichment;
- geometallurgical properties;
- possibilities for metal recovery;
- recommended reprocessing methods; and
- anticipated impacts of a site reintervention.

If a geoenvironmental assessment is also in the scope of the characterization, the assessments should include aspects, such as the likelihood of occurrence of waste waters, type of chemistry expected, impacts, and leaching pathways (GARD Guide, 2018).

The characterization of mine residues should be an interdisciplinary work that combines geology, mineralogy, geochemistry, hydrology, biology, and engineering. Ideally, the characterization works should be based on the definition of waste units that depend on the scope of the study and are supported by reliable legacy data. Furthermore, the standard analysis and test works (e.g., static and kinetic tests) should be complemented with low costs and rapid tests for screening the material on-site (Parbhakar-Fox & Lottermoser, 2015).

Even though there are inherent differences among mine waste storage facilities depending on, for example, the type of commodity, techniques for mineral processing, and regulatory framework, this chapter seeks to provide a general approach toward the sampling and characterization of mine residues in the view of CRMs recovery. Given the large variability of types of CRMs and the multiple types of mine residues where they can be possibly sourced (see Chapter 1), this work will address the bauxite residues as a case study. Nevertheless, other types of mine residuals will be considered when possible. In the following sections, we overview different sampling strategies and the techniques used to recover material by assessing their advantages and disadvantages. The characterization section focuses on analytical techniques rather than on test works. We present a selection of geochemical, mineralogical, and other techniques that can be used either in field environments or in the laboratory to study the main properties of the waste material. Overall, this review offers a tool for implementing better practices in the sampling and characterization for the revalorization of mine residues.

2.2 Critical raw materials in bauxite residues

The Bayer process is the standard method for beneficiating bauxite ores to recover alumina; this process uses an alkaline chemical attack with sodium hydroxide (NaOH). After the extraction of alumina, other components of bauxite considered impurities do not dissolve or are converted to sparsely soluble compounds disposed of as bauxite residuals, also sometimes referred to as red muds (Gräfe et al., 2011; Klauber et al., 2011; Power et al., 2011). Although the exact composition of bauxite residue depends on the type of bauxite and the process parameters of the Bayer process (Gräfe et al., 2011), an approximate mineral and chemical composition can be described in Tables 2−1 and 2−2, respectively. Secondary mineral phases formed during the Bayer process, such as hydrogarnet ($Ca_3(Al, Fe)_2(SiO_4)_y(OH)_{4(3-y)}$), cancrinite ($Na_6Ca_2CO_3$, $Al_6Si_6O_{24}\cdot 2H_2O$), perovskite ($CaTiO_3$), and gibbsite $Al(OH)_3$, are also present in the bauxite residues. Other factors influencing bauxite

Table 2–1 Mineral composition of bauxite residues.

Mineral	Chemical formula	Range (wt.%)
Hematite	α-Fe_2O_3	7–29
Goethite	α-FeOOH	7.3–24.3
Magnetite	Fe_3O_4	0–8
Diaspore	α-AlOOH	0.5–0.6
Boehmite	γ-AlOOH	1–9.6
Gibbsite	$Al(OH)_3$	1–5
Quartz	SiO_2	1.2–4.9
Rutile	TiO_2	1.1–5.4
Anatase	TiO_2	0.3–5
Sodalite	$Na_8[Al_6Si_6O_{24}][(OH)_2]$	2.7–24
Cancrinite	$Na_6[Al_6Si_6O_{24}]\cdot 2CaCO_3$	0–51
Calcite	$CaCO_3$	1–11.2
Perovskite	$CaTiO_3$	0–11.5
Kaolinite	$Al_4Si_4O_{10}(OH)_8$	Variable
Imogolite	$Al_2SiO_3(OH)_4$	Variable
Ilmenite	$FeTiO_3$	Variable
Hydrocalumite	$Ca_4Al_2(OH)_{12}$	Variable
Amorphous		Variable

Source: From Binnemans, K., Jones, P. T., Blanpain, B., Van Gerven, T., & Pontikes, Y. (2015). Towards zero-waste valorisation of rare-earth-containing industrial process residues: A critical review. *Journal of Cleaner Production*, *99*, 17–38. https://doi.org/10.1016/j.jclepro.2015.02.089; Gräfe, M., Power, G., & Klauber, C. (2011). Bauxite residue issues: III. Alkalinity and associated chemistry. *Hydrometallurgy*, *108*(1–2), 60–79. https://doi.org/10.1016/j.hydromet.2011.02.004.

Table 2–2 Chemical composition of bauxite residues from karstic bauxites determined by X-ray fluorescence (wt.%).

Bauxite origin	Refinery	Al_2O_3	Fe_2O_3	SiO_2	TiO_2	CaO	Na_2O
Turkey	Seydisehir	20.24	39.84	15.27	4.15	1.8	9.43
Turkey	Seydisehir	20.24	39.84	15.24	4.15	1.8	9.43
Ex Jugoslavia	Birac Alumina	14.14	48.5	11.53	5.42	3.96	7.5
Greece	Alumine de Grece	15.6	42.5	9.2	5.9	19.7	2.4
Turkey	Seydisehir	17.27	37.72	17.1	4.81	4.54	7.13
Greece	Alumine de Grece	15.85	48	6.96	7.06	14.84	3.26
Greece	None	16.94	39.34	6.95	4.79	13.2	2.1
Turkey	Seydisehir	19.1	37.6	15.7	4.9	2.4	9.5
Hungary	Ajka	14.8	42.1	13.5	5.2	6.1	8.9

Source: Adapted from Gräfe, M., Power, G., & Klauber, C. (2011). Bauxite residue issues: III. Alkalinity and associated chemistry. *Hydrometallurgy*, *108*(1–2), 60–79. https://doi.org/10.1016/j.hydromet.2011.02.004.

residue composition are NaOH content, heat and pressure conditions used during digestion, as well as chemical additives used (Liu et al., 2007). Karstic-type bauxites are richer in rare earth elements (REEs) in comparison with lateritic bauxites. The REEs are most likely to be associated with iron and titanium minerals that remain unchanged after digestion.

Even though the data available on the content of CRMs in bauxite residue are scant, some studies have indicated that the residues may be rich in valuable CRMs, such as scandium (Sc), vanadium (V), gallium (Ga), REEs, and other valuable elements [e.g., nickel (Ni), zinc (Zn), zircon (Zr), chromium (Cr)] (Binnemans et al., 2015; Deady et al., 2016; Deady, 2014; Ochsenkühn-Petropulu et al., 1994; Ujaczki et al., 2018). Furthermore, some studies report that bauxite residue is enriched in REEs compared to the bauxite ore by a factor of two (Ochsenkühn-Petropulu et al., 1994). This enrichment takes place due to the fact that (1) the extraction of alumina (Al_2O_3) leads to a concentration of other metals and (2) some processing plants combine ores from different origins (e.g., Mediterranean bauxites processed together with Caribbean bauxites). For example, the average concentration of REEs in bauxite from the Parnassos−Ghiona mountains in Greece is 506 ppm, whereas the average concentration of the resulting bauxite residue is 1040 ppm (Ochsenkühn-Petropulu et al., 1994). In addition, bauxite residues from two case study sites, one in Greece and the other in Turkey, have been found to contain an average of ∼1000 ppm of total REEs content, with higher prevalence of light rare earth elements (LREEs) over heavy rare earth elements (HREEs). Although this is relatively low grade compared with typical primary REE deposits (up to 80,000 ppm), it is of interest because of the large volumes available and the cost benefits of reprocessing waste (Deady et al., 2016).

Recovering scandium from bauxite residue is of high economic interest, as the metal represents more than 95% of the economic value of REEs in bauxite residue (Binnemans et al., 2015). However, there is little information available on the mineralogy of REE-bearing phases in bauxite residues. Furthermore, the nature and mechanisms causing variations in REEs distribution in bauxites are highly complicated, hindering understanding of the distribution and nature of the REE-bearing phases and minerals in the residues (Deady et al., 2016). Even though the association of REEs with iron and manganese phases is widely acknowledged in the literature, much more work is necessary to fully understand the distribution and mineralogy of REEs in bauxite residues. This will allow precise economic quantification of the potential REEs resources and could aid the development of new and more efficient leaching processes (Binnemans et al., 2015; Deady et al., 2016).

Bauxite residues might contain small quantities of radionuclides (from ^{232}Th and ^{238}U chains). Therefore they can be classified as Technologically Enhanced Naturally Occurring Radioactive Material (TENORM), falling under the legislation for Naturally-Occurring Radioactive Materials (Gu et al., 2012; O'connor et al., 2013). Because of this, legal constraints need to be considered when planning the intervention or repurposing of tailing sites.

Bauxite residues and red muds are solid solution mixtures ranging in initial solids content from 20% to 80% by weight, depending on the disposal method of the refinery. Bauxite residue consists of approximately 70% crystalline phases and 30% amorphous materials (Table 2−1). The mineral phases in bauxite residue are related to the content of the original bauxite ore (e.g., diaspore (AlO(OH)), boehmite (polymorph of g-AlO(OH)), gibbsite, Fe-oxides and hydroxides, kaolinite($Al_2Si_2O_5(OH)_4$)) and the secondary mineralizations that are formed during the Bayer process (e.g., perovskite, sodalite ($Na_4(Al_3Si_3)O_{12}Cl$), cancrinite, hydrated Ca-silicates, calcium aluminates, sodium carbonates) (Binnemans et al., 2015; Gräfe et al., 2011). CRMs in

bauxite residue are present in iron and titanium minerals (e.g., hematite, titanite, perovskite); LREE ferrotitanates are formed during the Bayer process digestion by an in situ transformation of the precursor bauxite LREE minerals contained in the bauxite feed. Minor amounts of LREE are found as carbonates and phosphates, referring to the partial resistance of these phases to Bayer process digestion conditions. HREEs are found in bauxite residue yttrium-phosphate phases (xenotime/churchite) (Vind, Malfliet, Bonomi, et al., 2018).

2.3 Sampling methods

The evaluation of the potential of reprocessing mine residues should be supported by credible information about the resources. This information can include historical records, but it must also contain a formal collection of data from the waste deposit (Bertrand, 2015; Muir et al., 2016). However, sampling mine residues is a challenging task. The compositional, spatial, and size heterogeneity of waste deposits makes using classical statistical methods problematic (Sädbom & Bäckström, 2018; Smith, 2000). The collection of samples and subsequent testing should be based on the specific objectives of a sampling campaign and should be well defined in the early stages of the project (Lapakko, 2002). The following section describes various types of physical collection of samples for mine waste rocks and tailings and the techniques that allow optimal recovery of sampling material.

2.3.1 Sampling strategies

When assessing the possible environmental impacts associated with mine wastes or the feasibility of reprocessing them, an exploratory or screening phase must be carried out to get an overview of the general characteristics of the deposit. Depending on the findings of this phase, a more detailed characterization can be performed for more precise identification of the recoverable resources. These differences also imply different approaches for sampling. It is also important to highlight that every mining site is unique, and site-specific information must always be considered to revise the sampling strategy.

The sampling of waste deposits will vary for waste rocks or tailings as their configuration and composition are different. Waste rocks are carried by dump trucks to the disposal site; the movement of the particles during the hauling process results in particle size segregation. Therefore waste rocks are composed of intercalated layers of coarse and fine-grained material. In contrast, for tailings storage facilities (TSF), the disposal methods depend upon the water content of the tailing and the topography of the tailings dam. The methods for material discharge include subaerial slurry, subaqueous slurry, paste, and dry deposition. The material in the tailings may also be segregated by grain size depending on the methods used at the latter stages of mineral processing (GARD Guide, 2018). Furthermore, the composition of the uppermost layers in a TSF might differ from the rest of the deposit since it has been exposed to (GARD Guide, 2018) enhanced weathering and leaching (Price, 2009).

When sampling waste rocks and tailings, it is crucial to ensure good representability of all geological materials present in the waste deposit. Therefore the number of samples must be

adequate to represent the total variability of the material. According to the GARD Guide (2018), ensuring representability entails:

- Compositional representation of all major materials based on the definition of geological units. Such units are few and robust during the screening phase, but that gradually increases detail and accuracy along with multiple sampling phases.
- Spatial representation in both the vertical and horizontal profile.
- Sampling method, that is, focussed versus random, depending on the specific objectives of the characterization program.

Price (2009) offered a comprehensive overview of the aspects that must be considered on the definition of sampling units, sample size, sampling storage, and preparation, among others applied to the characterization of drainage chemistry of sulfidic materials. However, most of the proposed protocols are equally applicable to evaluating the potential of reprocessing mine waste deposits. Hereby, we summarize some of those aspects.

2.3.1.1 Definition of geological units

To define the geological units and ascertain spatial representation, it is relevant to acquire legacy data of the waste deposit. This would include project type and history, geologic materials (excavated and processed), weathering environment (consider local and regional conditions), and mine components (methods of extraction and processing, rehandling, and secondary treatment). To ensure good geological and geochemical representation, a couple of samples collected using targeting sampling can be added within units with different physical, geochemical, and mineralogical properties. Ideally, the entire particle size distribution (PSD) should also be included among the material sampled.

2.3.1.2 Sample size

The area, volume, and length of individual samples should be based on the terrain's possibilities and the objectives of the sampling campaign. The sample size will depend on the spatial variability, the intended analyses and tests, and their requirements for accuracy and precision. During sampling, compositing samples must be avoided as it can mask properties specific to individual samples or result in misinterpretation of the results. Overall, it must be avoided to composite samples collected at different times, over vast distances, and from different geological units. Additional samples must be collected in the case of quality assurance and quality control assessments (QA/QC) or additional analyses and if the analyses are carried on different particle size fractions.

2.3.1.3 Sample storage and preparation

Appropriate sample storage must ensure that the geochemical conditions of the field are preserved (e.g., aerobic/anaerobic conditions). To avoid further alteration of the minerals, the samples must be air or oven dried at temperatures not higher than 40°C, or frozen if analyses of sulfide oxidation would be performed. During physical preparation, autogenous particle grinding should be avoided and the effects of sample preparation on the targeted

material properties should be considered. Likewise, the use of any chemical additives must be compatible with the subsequent analyses.

2.3.1.4 Reporting information

Although it might seem trivial, it is essential to highlight the relevance of good reporting practices during a sampling campaign. Organized and clear records regarding date, name of the samples, location, sample size (area, volume, length), type of material, project, and sampling technique are essential to conduct good analytical and test work and avoid misinterpretation of the results. In addition, it is helpful to keep a visual description of the samples and a photographic record of the samples and sampling conditions (e.g., the shape of the deposit, vegetation cover, vegetation type, etc.).

2.3.1.4.1 Sampling for the screening

The exploratory phase is used as a preliminary screen to assess the potential of a reprocessing project without any significant expenditure. The objective is to gather a general overview of the waste deposit and prioritize areas for further assessment. This phase does not produce any factual findings in terms of resource estimation, the viability of processing of the material, or plans for the mitigation of environmental impacts. The primary outcome from the exploratory phase is a recommendation on whether to proceed or abandon the reprocessing project (Muir et al., 2016).

At this stage, samples would be more likely collected using grab sampling, with special care on ensuring appropriate deposit coverage. For this, a sampling grid that enables the collection of 15−30 samples is recommended. To minimize the sampling errors, Smith (2000) proposed a method where the mine waste dump is divided into at least 30 cells of roughly equal surface area, and one sample is collected at the center of every cell. Sädbom and Bäckström (2018) suggest adapting the number of samples depending on the type of mine waste: often, tailings are more homogeneous than waste rocks and therefore would require a smaller amount of samples. For the design of the grid, these authors propose to set the grid-sample distance as follows:

$$\frac{\text{waste area}}{\text{number of samples}} = \text{area per sample} \tag{2-1}$$

$$\sqrt{\text{area per sample}} = \text{distance between samples} \tag{2-2}$$

For example, in the sampling of a tailings site with an area of 30,000 m^2 where 15 samples would be collected, the area per sample (cell size) would be 2000 m^2, and the sampling space $\sim$45 m.

Regarding the amount of material collected, Sädbom and Bäckström (2018) suggest that, given the higher heterogeneity of waste rocks, every waste rock sample must weigh 2−5 kg, whereas for the more homogeneous tailings, only 500 are required, always ensuring good representation of all particle sizes.

2.3.1.4.2 Sampling for detailed characterization

The sampling for detailed characterization seeks to explore the vertical profile, and therefore it is most likely to be conducted using drilling techniques. This type of sampling requires a more careful approach in the grid design using sampling theory (Lapakko, 2002). An optimal drilling grid defined using a geostatistical approach provides quantitative measures for resource estimation (Abzalov & Newman, 2017). In cases where the particle size is one of the targeted properties, attention must be paid that the chosen drilling technique can recover complete cores. For shallow sampling, trenches are recommended (Price, 2009).

Nevertheless, the sampling program must be designed with consideration of the site's characteristics and the resource material. According to the principle of 3D effective control on tailing dams, transects should be arranged at 50−200 m dot spacing, depending on the size and detail of the site (Muir et al., 2016; Pan et al., 2014). The discharge history, execution condition of the tailings dam, and geometry of the deposit should also be considered. Notably, an excessive number of sampling points next to the dam's walls or next to historical feed points should be avoided since they are a potential source of bias (Muir et al., 2016).

Samples collected for the detailed characterization of waste rocks are likely to be used for long-term field-based tests in cells with tens to hundreds of drill cores or large test pads with tones of mine rock material (Jamieson et al., 2015). In the latter case, collecting all particle sizes is a challenging task, particularly in the cases of open-pit mines where the particle size can reach up to 3 m. Therefore the sampling is restricted to particles smaller than 12 cm, being samples between 2 mm and 12 cm considered representative of the coarse size fraction (Price, 2009).

The samples collected for the detailed characterization of waste rocks and tailings usually undergo static or kinetic tests. The static tests are carried out on small subsamples of pulverized rock material (around 10 g). To ensure the representability of the subset sample, adequate techniques for sample preparation, handling, and splitting must be used. The samples used for kinetic tests are larger in volume, but only a few to tens of samples are required per study. The samples for such studies usually consist of coarsely crushed rocks for waste rocks test works, whereas for tailings the samples are simulated (Jamieson et al., 2015).

2.3.1.4.3 Sampling for determination of acid rock drainage and waste waters

One of the biggest concerns associated with managing mine wastes is the generation of vast amounts of poor-quality wastewater. The best-known type of wastewater is acid rock drainage (ARD), although mine drainage, in general, can be classified as acidic (pH 0−5), circumneutral (pH 6−8) or strongly basic (pH 8−12). It can also be radioactive, corrosive, and highly toxic (Nordstrom, 2011). ARD is produced through geochemical mineral weathering reactions that mobilize metals and metalloids (metal leaching) and other contaminants, causing severe environmental impacts (Jamieson et al., 2015). The mitigation of the impacts caused by ARD and metal leaching requires predictions of mineral reactivity and drainage quality to optimize waste storage conditions and effluent treatment.

In this context, the sampling and characterization of mine wastes also aim to provide accurate information about the intrinsic rock characteristics that can result in ARD or metal leaching (Jamieson, 2011; Parbhakar-Fox & Lottermoser, 2015; Price, 2009). For selecting a sampling method in this case, it must be taken into account that the target of the characterization is the detection of sulfides that are precursors of ARD, carbonate minerals that act as neutralizers, and problematic concentrations of metals and metalloids (Jamieson et al., 2015). In some cases, it is possible to use the same samples for both the evaluation of reprocessing potential of the waste and ARD and metal leaching determination. Attention must be paid that the methods for samples' handling and the intended test works are adapted to the aims of the study.

2.3.1.4.4 Sampling of mine residues using a geometallurgical approach

Most of the work developed around mine wastes focuses on the prediction of ARD formation. Thus it is not surprising that the sampling and characterization techniques for mine wastes are focused on identifying ARD precursors. Consequently, the practices for mine waste characterization most commonly follow the wheel approach proposed by (Morin & Hutt, 1995) that aims at classifying the waste rocks for predicting ARD risks. However, the scope of the work on mine wastes is broadened by the fact that mine residues are increasingly seen not just as an environmental liability but also as an opportunity for mineral recovery and revalorization. In that sense, the wheel approach has been criticized as it does not consider textural measurements and does not integrate geometallurgical data. In addition, other reference works, such as the Global ARD (GARD Guide, 2018), do not offer specific standards for sampling and characterization.

To improve ARD prediction and to offer valuable information for mineral recovery from mine wastes, mine residues should be studied more comprehensively. Following this principle, (Parbhakar-Fox, 2017) proposed an approach for characterizing mine residues that combines geochemistry, mineralogy, texture, and geometallurgy (GMTG). This approach focuses on the number of samples and the accountability for the material variability of the mine waste. For doing this, it is necessary to define not only geological units, as proposed by the (GARD Guide, 2018; Price, 2009), but also geometallurgical and geoenvironmental units, depending on the target of the study. The geometallurgical units are those intended for purposes of mineral recovery; they are ore type(s) with a unique texture, composition, and processes that result in similar metallurgical performances. The geoenvironmental units have similar textural, mineralogical, and chemical properties that control the generation of ARD and metal leaching (Parbhakar-Fox & Lottermoser, 2015).

In terms of sampling, the GMTG approach distinguishes between operational and historical sites. Following the recommendations of the (GARD Guide, 2018), the sample selection in operational sites must be based on the predefined units, and the samples must represent different size fractions of each type of material. For general characterization, the GMTG approach suggests collecting drill cores with 50 m of lateral distance and at least 50 m of depth to create a waste rock model. For detailed characterization, the size of the sampling grid should be between 2 and 50 m, depending on the variability of the mine waste deposit

(Parbhakar-Fox, 2017). For sampling of the historical sites, it is advised to collect 8−12 samples from each significant unit (assuming one unit accounts for approximately 2% of the total rock volume). Alternatively, collect one sample of 1.5 kg per 20,000 t of waste rock (i.e., 50 samples/Mt). Before these samples undergo conventional characterization (e.g., kinetic and static tests), they should be characterized, if possible, in situ. Information of interest, in this case is sample's geochemistry, mineralogy, and texture (Parbhakar-Fox, 2017).

2.3.2 Sampling techniques

The heterogeneity of the deposits is not the only challenge in mine residue sampling. Mine waste deposits are often unconsolidated material, which brings sampling issues, such as stability, sample contamination, and preservation. A better sample recovery is possible today, thanks to the recent developments, but the difficulty in accurately identifying these subsurface materials still remains a problem in some circumstances (Annels, 1991). The adequate assessment of CRMs in mine tailings requires high-quality samples that can provide a precise estimation of the metals' content and their distribution in the vertical profile. Among the plethora of sampling techniques, only those that permit high-quality sampling must be considered. Complementary to an adequate sampling technique, it is also necessary to count with well-trained operators who understand the importance of applying a rigorous procedure during sampling. Furthermore, the sampling theory must be followed with as much care as possible to reduce the losses due to poor sampling (Ortiz & Magri, 2014).

2.3.2.1 Grab sampling

Grab sampling is the simplest method for the collection of waste material. There is no specification for the equipment to be used, and the material is often collected in uncontrolled conditions. However, it is recommended to retrieve the samples with a stainless-steel trowel to avoid contamination and place them in a bucket upon collection. Air-dried samples are stored in a plastic bag or a paper soil-sampling bag to ensure complete drying of the sample (Smith, 2000). This sampling technique is only used for the screening of a waste deposit.

2.3.2.2 Trench sampling

It is one of the simplest methods for sampling unconsolidated material. A pit or a trench is dug by using a hydraulic excavator. This technique offers the advantage of exposing the sequence in situ and enables retrieving big particles (e.g., cobles) that cannot be recovered with drilling techniques. However, the excavation depth is limited by the water table and can cause substantial disturbance to the area (Annels, 1991). This method is common for shallow and dry deposits, or for obtaining bulk samples during a detailed investigation stage.

2.3.2.3 Auger drilling

Drilling using an auger is a widely used technique for obtaining samples of unconsolidated material like sand, gravel, or clays. It is popular due to the lower costs of operations: the equipment can be easily carried to the testing site and only one person is required to perform the

sampling (Annels, 1991). In this drilling system, the material is cut and broken with a simple blade bit mounted on the end of a rotating string of rods. Hand augers collect the material in a small barrel behind the bit, which is then pulled from the ground, and emptied when full. Screw or sleeved augers pass the material to the surface through a spiral screw thread along the rod string; this system is usually mounted on the back of a small truck or trailer (Marjoribanks, 2010). Fig. 2−1 shows the design of both hand and screw augers.

The depth limit for using hand augers is 2 m. Screw and sleeved augers reach greater depths (up to 45 m). The borehole is drilled and sampled in 1−1.5 m increments to provide suitable information on grade variation throughout the vertical profile of the waste deposit. The depth of the hole might be affected by the sand/slime stability, which is a function of the moisture content and particle size. After collection of every sample, the blades should be cleaned to avoid contamination (Muir et al., 2016).

2.3.2.4 Reverse circulation drilling

Reverse circulation (RC) drilling is a type of rotary percussion drilling. It has evolved due to the need to produce better quality samples, particularly below the water level (Annels, 1991). In this system, various rolled bits mounted on the end of a rotating string of rods cut and break the rock. High-pressure air pumped to the face of the bit down the center of the rods lubricates the cutting surfaces and helps to remove the broken material. In RC drills, the material is recovered through a separate tube inside the drill stem (Marjoribanks, 2010). Fig. 2−2 presents a diagram with the principles of RC drilling. RC rigs designed for drilling

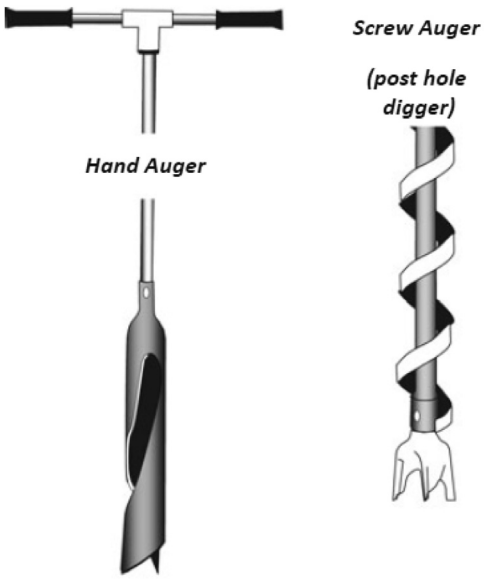

FIGURE 2–1 Diagram illustrating the main components of auger drills. *From Marjoribanks, R., & Marjoribanks, R. (2010).* Drilling: A general discussion the importance of drilling *(pp. 75−84). Springer Berlin Heidelberg. https://doi.org/10.1007/978-3-540-74375-0_5. Reproduced with permission from Springer Nature.*

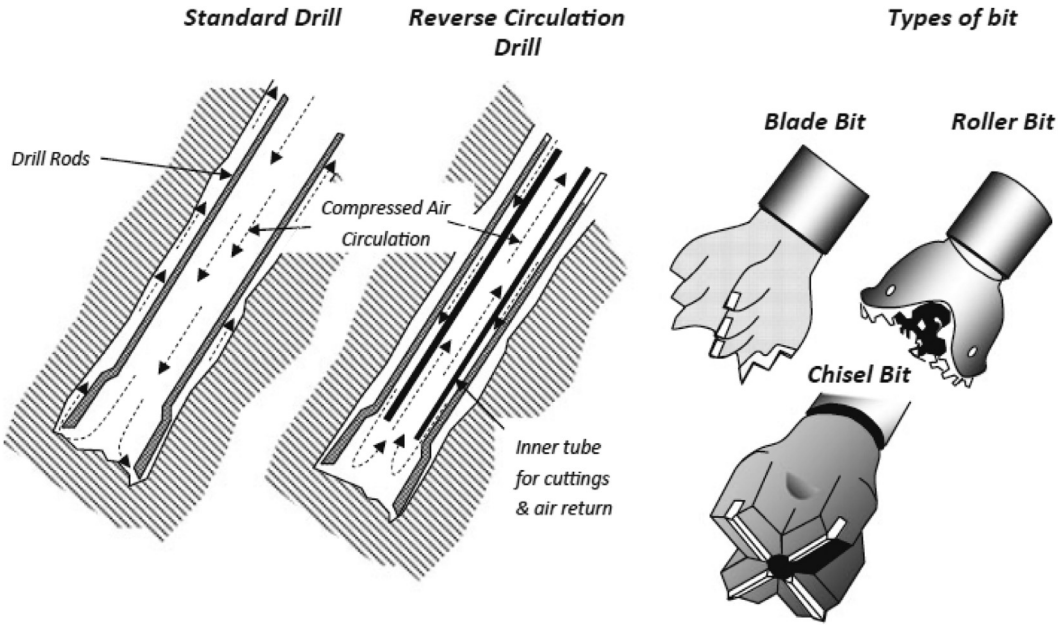

FIGURE 2–2 Diagram of the principles of RC drilling. *RC*, Reverse circulation. *From Marjoribanks, R., & Marjoribanks, R. (2010).* Drilling: A general discussion the importance of drilling *(pp. 75–84). Springer Berlin Heidelberg. https://doi.org/10.1007/978-3-540-74375-0_5. Reproduced with permission from Springer Nature.*

soft rocks usually use a smaller compressor and roller or blade instead of a tungsten carbide drill bit (Abzalov & Abzalov, 2016).

Unlike auger drilling, RC drilling recovers the entire sample, including any water, except for any cobles larger than the internal diameter of the rods (Annels, 1991). Contrary to augering, the continuous sampling eliminates the need to remove the drill string from the borehole. However, the fact that this system requires a compressor and additional equipment increases the costs of the operations. Therefore RC drilling is only recommended for detailed "second phase" drilling, where higher quality of samples is required.

2.3.2.5 Air coring

Air core is a type of specialized RC drilling that uses a small annular bit to cut a solid core of rock from relatively soft material. The bit produces short sections of core that are recovered in the same fashion as an RC rig. This system is most suitable for penetrating and coring soft, sticky clays, which may clog a standard blade bit (Marjoribanks, 2010).

2.3.2.6 Sonic drilling

Sonic drilling is a vibratory drilling method in which rotating weights from the drill rig's rotation unit send a vibration into the rod string and core barrel assembly. The sonic drilling method applies vibration to the drill string at a resonant frequency (typically between 50 and 120 Hz), which can fluidize particles adjacent to the drill string and significantly reduce

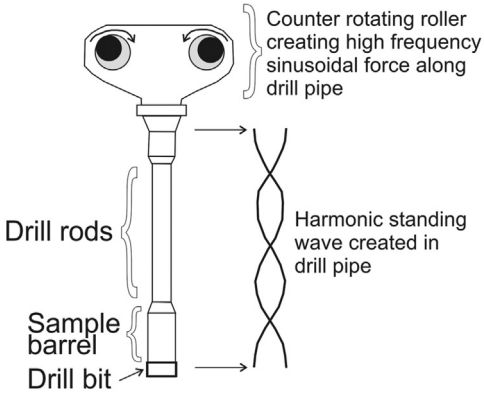

FIGURE 2–3 Diagram of the principles of Sonic drilling. *From Abzalov, M., & Abzalov, M. (2016).* Drilling techniques and drill holes logging *(pp. 39–77). Springer International Publishing. https://doi.org/10.1007/978-3-319-39264-6_4. Reproduced with permission from Springer Nature.*

friction (Fig. 2–3). This approach can provide rapid casing advancement down very straight holes with minimal or no fluid injection, produces relatively clean sidewalls and good core recovery, and generates minimal waste (Dinwiddie & Stothoff, 2013). Sonic drilling can be applied to relatively soft rocks and unconsolidated formations, including hard till and cobbles and boulders, and is particularly useful for monitoring tailings impoundments (Latimer, 2010). Sonic drilling is usually performed by track-mounted rigs allowing them free movement on unconsolidated sands and the soft clay formations (Abzalov & Abzalov, 2016). Even though this method is relatively costly, it might be cost-effective in terms of labor when numerous boreholes are required.

The sonic technique aims to collect representative undisturbed samples of nonconsolidated sands, which are essential for an accurate estimation of resources of the mineral sand deposits. Sonic drilling is the only technique that allows obtaining a continuous sample of the soft unlithified sedimentary formations, such as mine residues, in a cost-effective manner. As a result, sample integrity is significantly better than the other techniques, and the possibility of sample contamination is minimized (Abzalov & Abzalov, 2016).

2.3.3 Summary

Conducting an adequate sampling campaign is a critical step in the characterization of mine wastes. First and foremost, the general aims of the overall campaign or study must be clearly identified to select the most appropriate sampling strategies and, subsequently, sampling techniques. Next, with a clear scope, it is crucial to define the units (geological, geometallurgical, geoenvironmental) and the sampling scale (general/screening, detailed) and plan the analysis and test works that will be carried out. Finally, all these parameters set the basis for making decisions regarding grid density, number of samples, volume, duplicates, etc.

The selection of the proper technique or combination of techniques is usually a trade-off between speed, cost, required sample quality, sample volume, logistics, and environmental

Table 2–3 Drilling techniques for sampling mine waste deposits.

Drill type	Indications	Advantages	Disadvantages
Auger drilling	Geochemical sampling in the few upper meters of unconsolidated material	Human portable; contaminated mixed sample; quick; cheap	Poor penetration; sleeve augers depend on the power source
RC drilling	Geochemical sampling in hard and soft rock to 200 m plus	The sample might be contaminated; large sample volume; rock-chip composite returns (it loses context/textural relationships); relatively quick	Large, heavy rig or light-weight rig transported on the back of a 4 × 4 vehicle (requires an air compressor); no structural data; poor hole orientation control; some sample contamination/loss below the water table
Air coring	Geochemical sampling where good geochemical sampling and sampling of soft clays is required	Small rock-core return; minimal contamination, relatively quick	Small sample size; needs a source of air
Sonic drilling	Geochemical sampling; good for coring and monitoring	Accurate and continuous coring; minimal contamination; Ability to catch intact sample in unconsolidated material: preserves layering and fine scale textures.; no fluid dilution	Equipment is relatively costly

RC, Reverse circulation.

Source: Modified after Marjoribanks, R., & Marjoribanks, R. (2010). *Drilling: A general discussion the importance of drilling* (pp. 75–84). Springer Berlin Heidelberg. https://doi.org/10.1007/978-3-540-74375-0_5. Reproduced with permission from Springer Nature.

considerations. The specific requirements of the sampling program and the type of mine residue play an essential role in the choice of the drilling technique for detailed characterization. In addition, the level of consolidation of the material, amount of water in the waste deposit, and depth must be taken into consideration. Table 2–3 presents a summary of the main characteristics of every drilling technique presented here.

2.4 Analytical techniques for material characterization

To assess whether CRMs can be recovered from waste residues, it is essential first to assess the feasibility of such a project. Two main attributes must be considered at the initial stage: (1) determination of the presence of the target elements and (2) their association with the minerals in the deposit, which will determine the suitability of extraction. Using the concepts and methods of geometallurgy generally applied for standard mineral beneficiation is an excellent approach for mine residues (Dominy, 2018). It entails combining geochemical and mineralogical techniques to understand the composition of the waste material fully. Applying geometallurgy to the characterization of mine residues is embraced by the GMTG approach (Parbhakar-Fox, 2017). GMTG complements the traditional static and kinetic tests

(carried out mainly for ARD determination) with techniques that consider the texture of the material and its geometallurgical properties. It also emphasizes characterization techniques that can be used directly on the field to retrieve real-time information and aid in the (re)definition of geometallurgical and geoenvironmental units. Following the principles of geometallurgy, GMTG also stresses the importance of determining mineralogical properties as means of understanding mineralogical reactions that lead to leaching and metal mobility that might lead to metal enrichment or ARD generation (Parbhakar-Fox et al., 2018). The following section presents a review of chemical, mineralogical, and other supporting techniques for the detection of CRMs with emphasis on in-the-field techniques, as proposed by the GMTG approach, using the characterization of bauxite residue as an example of mine wastes.

2.4.1 Geochemical characterization

The determination of the presence of CRMs in bauxite residue is achieved through geochemical characterization. In the screening phase, the objective of the geochemical analysis is to indicate which CRMs are present in the tailings and give an initial estimation of the possible concentrations. Because of this, the techniques do not require high analytical sensitivity. The accuracy of this estimation varies depending upon the representability of the collected samples, particularly in the vertical profile, as metal concentration might be determined by element mobility. For resource estimation, that is, better quantification of the metal concentration in the tailings, it is necessary to use more specialized techniques with higher precision and sensitivity.

Many methods used for the geochemical characterization of mine residues are typically applied to rocks and soil samples. The analysis can be divided into destructive and nondestructive methods; destructive methods involve the dissolution of a solid sample (Jamieson et al., 2015). Among the instrumental methods available currently, X-ray fluorescence (XRF) spectrometry, laser-induced breakdown spectroscopy (LIBS), and inductively coupled plasma atomic emission spectroscopy (ICP-AES) have proven to be extremely useful. Nevertheless, instrumental neutron activation analysis (INAA) and inductively coupled plasma mass spectrometry (ICP-MS) are commonly used for REE determination in different kinds of materials (Balaram, 2019). The following section presents a description of these techniques, and Table 2−4 presents a comparison of their performance based on the detection limit for some CRMs.

2.4.1.1 X-ray fluorescence

XRF is a well-established technique for qualitative and quantitative analysis of most chemical elements (excluding the lightest ones). An X-ray tube is used to generate secondary X-rays emissions from a particular element in a sample; the intensity of the emissions is proportional to the concentration of the element in the radiated area. Bench units are commonly used in laboratory settings, whereas portable hand-held units (pXRF) are suitable for field applications (Jamieson et al., 2015; Lemière, 2018; Bodénan et al., 2015). The detection limits are often higher than for the other analytical methods (Table 2−4), with the portable units having lower sensitivity than the bench ones. Portable instruments are most useful as rapid

Table 2–4 Detection limits of rare earth elements and Sc obtainable by some popular instrumental analytical techniques.

	Element	XRF (μg/g)	LIBS (μg/g)	INAA (μg/g)	ICP-AES (μg/g)	ICP-MS (ng/mL)
LREEs	La	10.10	10.00	0.19	0.50	12.74
	Ce	22.10	–	0.03	0.10	0.61
	Pr	9.10	40.00	–	0.02	0.72
	Nd	1.80	500.00	3.03	1.00	2.45
	Sm	8.20	40.00	0.08	1.30	0.98
HREEs	Eu	–	5.00	0.03	2.60	0.37
	Gd	7.10	200.00	–	0.50	0.98
	Tb	–	60.00	0.10	0.70	0.07
	Dy	9.30	10.00	–	0.60	1.41
	Ho	–	–	–	0.80	0.07
	Er	–	30.00	–	0.10	0.11
	Tm	–	30.00	–	0.08	0.37
	Yb	–	–	0.08	1.60	0.37
	Lu	–	20.00	0.04	0.40	0.02
	Y	6.40	–	–	0.80	3.68
	Sc	–	2.00	–	0.05	11.03

HREEs, Heavy rare earth elements; *ICP-AES*, inductively coupled plasma atomic emission spectroscopy; *ICP-MS*, inductively coupled plasma mass spectrometry; *INAA*, instrumental neutron activation analysis; *LIBS*, laser-induced breakdown spectroscopy; *LREEs*, light rare earth elements; *XRF*, X-ray fluorescence.

Source: From Balaram, V. (2019). Rare earth elements: A review of applications, occurrence, exploration, analysis, recycling, and environmental impact. *Geoscience Frontiers*, *10*(4), 1285–1303. https://doi.org/10.1016/j.gsf.2018.12.005. Creative Commons license.

screening tools to identify enriched areas or assist in designing a sampling program for detailed analyses (ThermoFisher, 2017b). New technological developments quickly improve the number of detected elements, detection limits, and data acquisition speeds.

CRMs, particularly REEs, have been successfully identified in Greek bauxite residue using XRF (Borra et al., 2016; Ochsenkühn-Petropulu et al., 1994). The reported values are in the magnitude of the tens and hundreds of ppm. Even though XRF is a reliable technique, it is relatively insensitive to the REEs; nevertheless, it has advantages over other methods regarding accuracy, speed, and costs. Because of the high detection limits, many determination methods involve separation and preconcentration procedures to provide an accurate determination (Balaram, 2019). Recently portable XRF is being successfully used for on-site field quantification of REEs, including lanthanum (La), cerium (Ce), praseodymium (Pr), and neodymium (Nd), and typical REE pathfinders in geochemical exploration studies, including yttrium (Y), thallium (Th), and niobium (Nb) (Balaram, 2017). However, a recent study conducted by Gallhofer and Lottermoser (2018) showed that the spectra collected from certified reference materials presented poor accuracy and false positives associated with spectral interferences. Critical elements may be determined in common geological materials when pronounced peaks occur in the spectra and that matrix match of standards and samples is essential. Hence, XRF spectra should be routinely reviewed to identify erroneous quantification due to spectral interferences.

XRF, and in particular pXRF, has been used successfully, for example, for in situ sampling analysis of waste rocks from Pb−Ag mining (Bertrand, 2015). In this case, pXRF was used for chemical screening analyses at the dump surface and on grab samples. The pXRF results demonstrated the homogeneity of waste piles from similar beneficiation schemes, which influenced the strategy for metal recovery. In the same study, the tailings from a former tungsten mine were also analyzed; the on-site pXRF results enabled the identification of two facies along with a redox profile by analyzing the tungsten (W), copper (Cu), and arsenic (As) contents.

2.4.1.2 Laser-induced breakdown spectroscopy

LIBS is a nondestructive technique that utilizes a pulsed laser beam to ablate a small amount of material on the surface of a sample and break it down into a plasma consisting of atoms, ions, and free electrons. The chemical composition of a material can be characterized by measuring the radiative emission of the plasma with a spectrometer. The amount of detected elements with this technique is more extensive than with XRF. Even though LIBS can be used for both qualitative and quantitative characterization, differences in sample composition, crystallinity, density, grain size, hardness, and surface roughness make the careful calibration of an LIBS system an essential requirement for quantitative compositional analyses (Harmon et al., 2013; Yaroshchyk et al., 2010). LIBS sensor systems currently available include laboratory instruments, hand-held instruments (Lawley et al., 2021), and conveyor belt scanners (Laser distance spectrometry, 2017; Secopta, 2017; Sonicsampdrill, 2017). The most significant advantage of LIBS is its ability to make real-time identification of different metals, including REEs and non-metals, in seconds (Bhatt et al., 2018).

Some CRMs, including REEs, have been identified in geological samples using LIBS (Bhatt et al., 2018). Detection limits can range between 10 and 100 mg/g for most REEs in common applications (Table 2−4), with precisions generally ranging from 3% to 5%. For homogeneous materials, an error of less than 2% can be achieved (Balaram, 2019). Bauxite ores have also been characterized using this technique (Fahad et al., 2019).

Kuhn et al. (2016) developed a prototype of a core scanner working with LIBS to estimate the presence of metals in mine tailings. By doing elemental mapping, the researchers could draw conclusions related to the distribution of the metal-bearing minerals and to identify the zones with elevated metal concentrations. Using a similar instrumental set-up, (Müller et al., 2021) detected REE-rich areas in highly heterogeneous geological materials. LIBS enabled the spatial and geochemical analysis of the REE-bearing minerals in the analyzed samples.

2.4.1.3 Instrumental neutron activation analysis

INAA is a versatile and highly sensitive technique for detecting the concentration of major, minor, and trace elements. The sample is subjected to a neutron flux in a nuclear reactor where the stable nuclei absorb neutrons and become unstable radioactive nuclides that decay, generating gamma rays. The energy emitted by the gamma rays is characteristic of the elements that generated it, and the intensity can be used to determine their abundance. This technique is prevalent because nuclear reactions and decay processes are virtually

unaffected by the chemical and physical structure of the material during and after irradiation. Several workers have applied the INAA technique to determine REEs at extremely low concentrations in different earth and environmental samples (Table 2−4). For precise REEs determination, it is necessary to use synthetic standards that match the sample matrix. Despite its precision, INAA is not a popular analytical technique as it is time-consuming, not independent, requires a reactor nearby, and involves longer cooling times for certain elements (Balaram, 2019).

Several researchers have applied the INAA technique to determine REEs at extremely low concentrations in different earth and environmental samples. For example, Vukotić (1983) used INAA for detecting REEs [except for Pr and erbium (Er)] in different types of bauxites. Anawar et al. (2012) assessed REEs bio-accumulated in tailings from a copper-sulfide area, finding differences in enrichment between LREEs and HREEs in lichens.

2.4.1.4 Inductively coupled plasma atomic emission spectroscopy and inductively coupled plasma mass spectrometry

There are several techniques based on inductively coupled plasma (ICP) spectrometry. These are destructive techniques based on the ionization of the sampled material after digestion in an appropriate medium. If a complete elemental analysis is desired, total or near-total dissolution by strong acid mixtures is the typical course, followed by the analysis of the resulting solution by ICP (Jamieson et al., 2015).

One of the ICP techniques is ICP-AES, also known as ICP Optical Emission Spectroscopy (ICP-OES). In this technique, plasma ionization produces excited atoms that emit electromagnetic radiation at element-characteristic wavelengths as they return to ground state. ICP-AES can measure up to 60 elements simultaneously with high sensitivity (Table 2−4). However, to determine REEs in geologic materials, it is necessary to separate and preconcentrate the samples (e.g., by precipitation, solvent extraction, and ion exchange) before the measuring procedure to avoid spectral interferences (Balaram, 2019).

ICP-MS is a form of ICP with excellent sensitivity, minimal interferences, good precision, and accuracy (Balaram, 2019). The technique relies on linking the ICP to a mass quadrupole spectrometer; the ions are separated based on their mass-to-charge ratio, and a detector receives an ion signal proportional to the concentration. ICP-MS is an excellent analytical technique due to its extremely low detection limits (Table 2−4), high sample throughput, the requirement of minimal quantities, element versatility (major, minor, trace, and ultra-trace), and isotopic detection (Balaram et al., 1995). Currently, the ICP-MS technique is being extensively applied for the accurate determination of REEs in different types of materials (Balaram, 2019).

2.4.2 Mineralogical characterization

Mine residues present a higher diversity of mineral phases than the naturally occurring materials at the Earth's surface. This makes it more challenging to identify and quantify minerals in mine wastes (Jamieson et al., 2015). Nevertheless, the recovery assessment for

CRMs in mine wastes, such as bauxite residue, should also involve the mineralogical characterization of the material. Besides determining the presence of CRMs, it is necessary to elucidate how the metals are hosted in the bauxite residue, that is to say, find their relationship with the minerals. This information is essential for understanding the mobility of the metals —which affects their spatial distribution—and for selecting the techniques for the recovery and processing of the CRMs.

In the case of bauxite residues, the mineralogical characterization should involve techniques that can detect the residual minerals from the beneficiation process, the newly formed minerals as a consequence of the Bayer process, and the amorphous phases (Table 2−1). Likewise, the geochemical techniques, the techniques for mineral characterization can be nondestructive or destructive and have varying levels of accuracy. Some of the techniques for the mineral characterization of mine wastes include X-ray diffraction (XRD), different forms of infrared (IR) spectroscopy, and Raman spectroscopy.

2.4.2.1 Infrared spectroscopy

IR reflectance spectroscopy has become a popular technique among geochemists and mineralogists since it is relatively rapid, inexpensive, and requires little or no sample preparation. As a consequence, IR is becoming increasingly established in the mining industry in applications, such as mining monitoring (Guatame-Garcia & Buxton, 2020; Kirsch et al., 2018), ore sorting (Dalm et al., 2017), and characterization of slurries (Haavisto & Hyötyniemi, 2011). IR is used to determine the mineral composition, bonding of the molecules, and mineral structure. In this way, it is possible to identify minerals based on their functional groups, for example, clay minerals (OH), carbonate minerals (CO_3), and silicates (SiO_4) (Farmer, 1974). In addition, the enhanced sensitivity of modern spectrometers and the versatility of the sampling capabilities have facilitated the development of laboratory and field instruments. More interestingly, they have opened the possibility of making direct interpolations between these environments (Madejová et al., 2011).

Depending on the application and instrumentation, the IR spectrum is recorded in frequency or wavelength units. For laboratory applications, frequencies are expressed in wavenumbers (cm^{-1}), whereas for remote sensing, hand-held, and online applications, it is common to use wavelength expressed in micrometers (μm). For convenience in instrumentation and sampling, the IR spectrum is split into ranges depending upon the application field and the units' usage. Since this review is oriented toward mining and processing on-site applications, the remote sensing, hand-held, and online convention is used. In this sense, the units appear in wavelengths, and the spectral ranges are defined as visible−near IR (VNIR), short-wave IR (SWIR), mid-wave IR (MWIR), and long-wave IR (LWIR). Table 2−5 presents the wavelength and wavenumber equivalent for these spectral ranges.

The main mineral features occur in the LWIR and MWIR ranges. Most of them are a consequence of the metal−oxygen (M−O) stretching modes and the metal−hydroxyl (M−OH) bending and lattice modes. On the other hand, the secondary features are strongly present in the SWIR range and involve mainly OH groups. All these spectral features make possible the identification of a significant portion of the mineral species (Hunt, 1977; Madejová et al., 2011).

Table 2–5 Equivalent wavelengths and wavenumbers of the main spectral ranges.

Spectral absorption band or range	Wavelength (μm)	Wavenumber (cm^{-1})
VNIR range	0.35–1.00	28,571–10,000
SWIR range	1.00–2.50	10,000–4000
MWIR range	2.50–7.00	4000–1429
LWIR range	7.00–16.00	1429–625

LWIR, Long-wave infrared; *MWIR*, mid-wave infrared; *SWIR*, short-wave infrared; *VNIR*, visible–near infrared.

The recent technological advances and the growing demand for IR spectroscopy have increased the affordability of IR instruments. The technological improvements are especially moving toward analyzing the sample "as is," minimizing or avoiding any sample preparation, and shortening the time required to record the spectra (Chryssikos & Gates, 2017).

The potential applications of IR instruments also depend on the type of sampling needed to analyze a particular material. Some instruments require the preparation of KBr pellets or thin films to enhance the signal, particularly in the MWIR and LWIR ranges. Other instruments allow the use of pressed powders or solid specimens instead, which is more suitable for geological materials. Instruments designed to be used in field applications allow the use of samples with little or no preparation, that is to say, powders and solids. In these cases, the spectral response can vary according to samples' characteristics, such as grain size, surface roughness, and porosity (Rost et al., 2018; Salisbury & Wald, 1992).

Different modes of analysis are possible depending on the recording geometry of the instruments. According to King et al. (2004), the most frequently used geometries for geological applications are attenuated total reflectance (ATR), specular reflectance, and diffuse reflectance. ATR is perhaps the most used method for laboratory characterization. The ATR uses a special prism to produce internal reflections from the surface of a given material. Specular reflectance, also known as bidirectional or external reflectance, uses an incident IR beam at an air/sample interface; the intensity of the reflected beam depends on the direction of the incident energy and the surface characteristics of the sample. Diffuse reflectance measures the scattered and reflected light diffusely, especially from loose powders, requiring minimum or no sample preparation. The instrument can also integrate an interferometer that collects the so-called Fourier-transform IR (FTIR) spectra.

2.4.2.1.1 Visible–near infrared and short-wave infrared spectral reflectance

Spectrometers, such as the pioneer portable IR mineral analyser (PIMA) or the analytical spectral devices (ASD) FieldSpec, have been used since the 1990s. Nowadays, they are well known as an important mineral characterization tool for geologists and mineralogists (Thompson et al., 1999). These are dispersive instruments that record point data in the VNIR and SWIR ranges. Following the same principle, there has been significant development of hyperspectral imaging systems to analyze drill cores and hand samples. Examples of this technology are the SisuROCK hyperspectral core logger (SPECIM) and the Hylogger system (CSIRO) (Schodlok et al., 2016). Similar observations can be made using remote sensing

airborne and satellite images. These techniques have been used, for example, in the mapping of areas with bauxite outcrops or for the monitoring of red muds (Ibrahim et al., 2018; Marion & Carrère, 2018).

Hyperspectral core scanning images have been extensively used for mineral exploration and resource estimation (Dalm et al., 2017; Fox et al., 2017; van Ruitenbeek et al., 2019). Surprisingly, their use in the characterization of mine wastes has started to develop only recently. While the focus so far has been on the environmental aspects related to the generation of ARD (Cracknell et al., 2018), there is a growing interest in the use of this technology for the prospectivity of critical metals in mine wastes (Hubbard, 2020).

Reflectance spectroscopy in the VNIR and SWIR ranges uses specular reflectance and can detect several of the minerals present in bauxite red muds. The iron-oxide reflectance spectrum strongly reflects the visible and near-IR region due to various electronic transitions (Hunt, 1977). Specifically, goethite features occur due to ion charge transfer at 0.50 μm and iron-oxide at 0.95 μm (Ramanaidou et al., 2015). The bauxite ore minerals have distinctive absorption features in the SWIR range due to the hydroxyl molecule (Hunt, 1977). REE-bearing compounds are known to produce sharp absorption features in the VNIR to SWIR regions. Exploitable spectral differences for identifying REE-bearing fluorocarbonates include a distinct absorption band at 2.243 μm and the one around 1.965 μm, which separate different mineral species (Turner et al., 2014).

2.4.2.1.2 Mid-wave infrared and long-wave infrared spectral reflectance

Since the main spectral features appear in the MWIR and LWIR regions, many overlaps are solved. Absorption bands between 8 and 12 μm might allow a better differentiation among REE-bearing phosphates (Laakso et al., 2019). These regions can, in many cases, enable better differentiation of the bauxite ore minerals and the amorphous phases present in the bauxite residue. Identifying oxide minerals in the MWIR and LWIR ranges is not possible as they do not present any features in this region.

Measurements with high spectral resolution and high signal-to-noise ratio can be acquired with laboratory setups that use ATR or FTIR spectrometers. These can be classified as semidestructive methods since they require KBr pellets or powder samples. Recent technological developments have made it possible to use FITR spectrometers in portable devices that retrieve point data from rock samples. It is also possible to acquire hyperspectral images in the LWIR range using the same imagers as described above, although with lower resolution (spatial and spectral).

Bauxite ore minerals and associated Fe and Ti phases have been successfully characterized in laboratory environments using ATR and FTIR spectroscopy (Ruan, 2005). FTIR portable devices have been used to monitor the quality of bauxite ores in mineral production environments (Eyers, 1999; McGuiness, 2005). Aluminum phases have also been identified with this same type of instruments (Guatame-Garcia & Buxton, 2018). Drill core scanning of mine wastes in the LWIR range (also known as thermal IR—TIR—or IR thermography) to detect and visualize the temperature response of sulfide oxidation (Knobloch & Lottermoser, 2020).

Even though clear absorptions in the VNIR and SWIR ranges cannot be confused with those of other minerals, the presence of iron oxides, active in the same wavelength range, can hamper the detection of the REEs even in small quantities (Boesche et al., 2015). Moreover, the spectral features of different REEs overlap both in the VNIR and SWIR wavelength regions, making it challenging to identify with certainty the presence of a specific rare earth element in a mineral in many cases (Turner et al., 2014, 2016).

2.4.2.2 Raman spectroscopy

Raman technology is used to record the Raman scattering that materials produce due to the photon emissions caused by molecular vibrations. Raman scattering can be measured by illuminating a material with a monochromatic light source and measuring the emission spectrum with a spectrometer. The Rayleigh scattering is dependent on the wavelength of the light source, which is commonly a laser. However, the Raman scattering is independent of the wavelength of the light source; therefore almost any type of laser can be used. Raman sensors are mainly used for qualitative analysis (Gaft, 2005). Quantitative analysis is possible but requires careful calibration.

Laboratory and hand-held instruments are available for Raman analyses (Sciaps, 2017; ThermoFisher, 2017a). Point data and microscopic images are well-established methods for the collection of Raman measurements. More recently, applications for drill-core imaging have been emerging (Wells & Ramanaidou, 2015).

Raman spectroscopy can be used to detect CRM-related minerals. For example, it has shown promising results in the characterization of Fe-bearing minerals (Wells & Ramanaidou, 2015) and bauxite ores (Faulstich et al., 2011; Ruan et al., 2001). In addition, the artifacts in the Raman spectrum are being used for identifying REEs using Raman spectroscopy (Lenz et al., 2015).

2.4.2.3 X-ray diffraction

XRD is a nondestructive technique that provides detailed information about the crystal structure, chemical composition, and physical properties of materials. First, the sample is irradiated with incident X-rays, then the intensity and the angle of the scattered X-rays are measured. Then, the interpretation of the mineralogy and structure of the sample is made by matching the diffraction patterns against databases with patterns from several mineral and industrial compounds.

XRD is a well-established technique that has been widely applied to characterize mine waste mineralogy (Jamieson et al., 2015). However, to generate quantitative mineralogical data from multiphase mixtures, as is the case of mine tailings, it is necessary to use the Rietveld method for crystal structure refinement (Rietveld, 1969). Furthermore, the complexity of the mineral assemblages in bauxite residues makes challenging the identification of clear diffraction peaks necessary for mineral identification. Moreover, while some mineral phases might be present at high concentrations, others are present as solid solutions that cannot be matched with a database. All these issues can be overcome by using the Rietveld method (Santini, 2015):

- rietveld refinement-based methods quantify multiple minerals present in a sample simultaneously;
- accommodate nonideal mineral compositions (due to isomorphous substitution of Al for Fe in iron oxides);
- correct for other sample-related effects on peak shape and area, such as particle size and preferred orientation;
- allow quantification of X-ray amorphous content (which can be particularly useful for geochemical modeling and process optimization).

2.4.3 Microanalysis

Since mine waste samples are made of mineral mixtures, their characterization should also involve the analysis of individual grains. Furthermore, textural analysis at the microscale is also required to identify the mineralogical hosts of the environmental and economic elements of interest, detect the mobility of elements, and understand the reactivity and development of secondary mineral phases. For this kind of analysis, it is common to prepare thin sections (Jamieson et al., 2015). When a thin section is bombarded with electrons or protons to generate X-rays, then the corresponding analytical method is called scanning electron microscopy (SEM) or electron probe micro analysis (EPMA) (Terzano et al., 2019). SEM provides high-resolution images rather than quantitative analyses. In contrast, EPMA is optimized to collect quantitative data in concentrations as low as a few hundred ppm (Jamieson et al., 2015); however, the minimum particle size that EMPA can analyze is 1 μm (Vind, Malfliet, Blanpain, et al., 2018).

The combination of high-resolution microscopic images obtained with SEM and analysis with software, such as QEMSCAN (Quantitative Evaluation of Minerals by using SCANning electron microscopy) and MLA (mineral liberation analysis), is advantageous in the characterization of fine-grained materials, such as tailings, soil, and contaminated sediments. Because of the high resolution, individual minerals can be characterized, enabling a particle-by-particle characterization, including chemical composition, shape, and size (Jamieson et al., 2015).

2.4.4 Other techniques

Another essential aspect in the characterization of mine residues is the evaluation of individual grains. Their size, shape, and texture greatly influence the chemical and mineral attributes and therefore the most feasible methods for their characterization. Furthermore, some of these properties are highly relevant to the selection of residue reprocessing strategies. The methods used for the characterization of particles in mine wastes are often those used in soil testing. The most common physical analyses are PSD, specific surface area (SSA), and specific gravity (SG). Several microanalysis techniques for the mineralogical characterization of individual grains are also widely used. Other techniques for evaluating a mine waste deposit as a whole include gamma-ray spectroscopy and fluid chemistry.

2.4.4.1 Laser granulometry

The particle size of bauxite residues averages 2−100 μm with a typical range of 100 nm to 200 μm (Gräfe et al., 2011). However, the particle size and texture of the residues depend on the location within the disposal area (Gräfe et al., 2011; Nikraz et al., 2007). These variations influence chemical parameters, such as pH and Na, Ca, and Mg concentrations (Fig. 2−4). Since most of the particles consist of silt and clay, one of the most common methods for measuring the PSD is wet sieving. Particles smaller than 200 μm can be further studied using hydrometer analyses. For the smallest particles, methods, such as turbidimetry and electron microscopy, are recommended. However, an alternative and more efficient method is the laser particle size analyzer.

Laser granulometry, or laser diffraction, is commonly used to determine PSD. This technique can measure particles as small as hundreds of nanometers up to several millimeters (Malvern Panalytical, 2021). When circular or spherical particles are illuminated with a laser beam, they scatter the light into a certain angle, which is the basis for particle size determination (Heuer & Leschonski, 1985). Large particles scatter light at small angles, whereas small particles scatter light at large angles. While the scattering angle is only a function of the particle size, the scattering intensity is a function of the particle size, the refraction indices of the particle and the dispersant, the wavelength of the incident radiation, and the scattering angle (Dur et al., 2004; Malvern Panalytical, 2021).

The laser diffraction technique uses the Mie theory of light scattering to calculate the PSD. For small particles (<1 μm), the calculations are based on the Rayleigh−Debye−Gans theory, whereas for particles larger than the incident wavelength (∼ >1 μm), the Fraunhofer theory is more appropriate. Rayleigh−Debye−Gans and Fraunhofer's theories require determining the refractive index of the solid phases and the dispersant (Dur et al., 2004). However, for particles larger than 50 μm, it is possible to use the Fraunhofer approximation, which does not require any knowledge of the sample's optical properties (Malvern Panalytical, 2021). Regardless of whether the particles are stationary or moving, the scattering pattern remains the same. However, under- or overestimations occur for platy-shaped

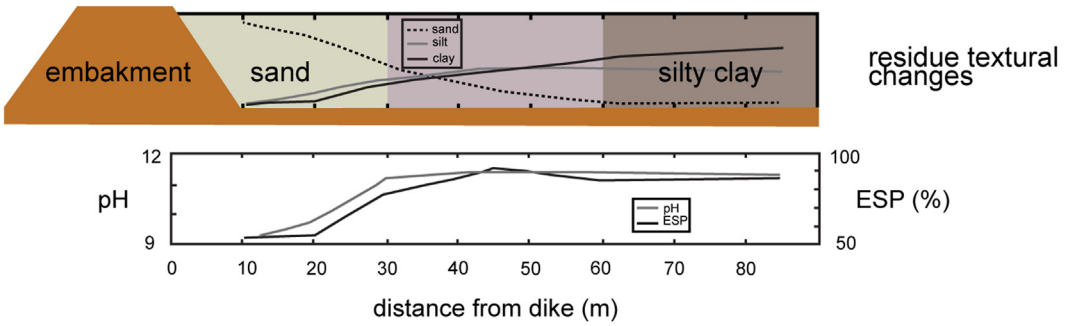

FIGURE 2–4 Particle size variations along a red mud disposal area and their influence on pH. *From Gräfe, M., Power, G., & Klauber, C. (2011). Bauxite residue issues: III. Alkalinity and associated chemistry.* Hydrometallurgy, 108*(1−2), 60−79. https://doi.org/10.1016/j.hydromet.2011.02.004. Reproduced with permission.*

particles as the calculations assume that the particles are spherical. This is particularly the case of clay minerals (Gorączko & Topoliński, 2020). The PSD calculated with laser diffraction is expressed in volume units, surface area units, or number of particles (Dur et al., 2004; Malvern Panalytical, 2021).

Laser granulometry presents several advantages compared to other PSD techniques. First of all, this technique outperforms other particle size measurement techniques in speed, reproducibility, and simple handling (Heuer & Leschonski, 1985). Second, laser granulometry can size very fine particles compared to other methods (e.g., sieving, sedimentation) (Dur et al., 2004). In the characterization of tailings, laser diffraction has become one of the preferred techniques for determining particle and aggregate size distributions. For example, Jiangang et al. (2012) presented a case study of reprocessing of ultrafine molybdenite tailings. Laser granulometry was used to study the original mineral particles and agglomerates to optimize the parameters for the recovery of residual ore. In a similar case, Dubos et al. (2018) used laser diffraction in industrial manganese dust and sludges to evaluate critical parameters to design appropriate agglomeration processes.

2.4.4.2 Techniques for the determination of specific surface area and specific gravity

The average **SSA** of bauxite residues is 32.7 ± 12.2 m^2/g and ranges between 15 and 58 m^2/g (Gräfe et al., 2011). The SSA is defined as the total surface area of a material per unit of mass. There are several methods for determining SSA, which can be classified into methods based on the adsorption of gas or the adsorption of polar liquids. The gas adsorption methods are deemed as a measure of the external surface area, whereas the polar liquid methods also account for the internal surface area (e.g., micropores, interlayer space) (Heister, 2014). The most frequently used methods for SSA determination are the physisorption of nitrogen gas at 77K (BET-N_2) (adsorption of gas method) (Fagerlund, 1973) and the retention of ethylene glycol monoethyl ether (adsorption of polar liquids method) (Gregg et al., 1967).

In soil science, **SG** refers to the ratio of the density of the solid's volume to the density of any equal volume of water at 4°C. It is conventionally determined using the He pycnometer method for coarse-grained materials or the density bottle method for materials of all grain sizes (ASTM, 2007). An alternative method for fine-grained materials is the shrinkage test procedure (Prakash et al., 2012).

2.4.4.3 Techniques for determination of mineral hardness

The hardness of a material is a property that measures the susceptibility to breaking. In the characterization of mine wastes, this property can be one of the indicators of breaking behavior in the case of mineral reprocessing or how quickly acid minerals become exposed to weathering and cause ARD. Most of the existing techniques are destructive and require sample preparation procedures. However, the EQUOtip rebound hardness test (Keeney & Nguyen, 2014) is not destructive and can be used on drill-core samples. It measures the material's hardness based on a calculation of the speed of the impact rebounds of the probe over a sample. The hardness criterion is site-specific and must be defined based on test work (Parbhakar-Fox, 2017).

2.4.4.4 Fluid chemistry

The properties of the particles present in mine waste deposits (i.e., the solid interface) are also influenced by the interactions with the fluids that circulate through the deposit (i.e., the liquid interface). Therefore it is also important to use fluid chemistry to obtain a comprehensive overview of the entire deposit. The measurements of the bauxite residue alkalinity and associated physicochemical characteristics are carried out with analysis, such as pH, acid-neutralizing capacity (ANC), sodium (Na^+), electrical conductivity (EC), and surface charge (Gräfe et al., 2011).

Many researchers consider the measurement of pH as the master variable because most reactions are, in a way, a function of pH. Measuring pH is a simple technique conducted in the field or the lab using pH meters. The **ANC**, also known as alkalinity, measures the amount of mineral acid required to reach a specific pH point. This ANC can be best measured in the laboratory by a process known as back titration. The method involves dissolving the antacid in excess of acid and then titrating the acidic solution against a known base concentration until the endpoint is reached. Detection of $\mathbf{Na^+}$ in clay–water mixtures is relevant since it can be related to the total alkalinity of red mud, poor aggregate structure, cementation, and dust formation. High concentrations of Na^+ result in high EC, which relates to physical behavior, such as dispersion and coagulation. Conductivity is measured with a probe and a meter: voltage is applied between two electrodes in a probe immersed in the sample water. The inter- and intraparticle behavior of waste material in the presence of water depends on the particles' surface charge. This affects not only gross macroscopic physical behavior, such as rheology, aggregation, and coagulation, but also the entire range of particle chemistry. The surface charge is measured in the laboratory using the so-called zeta-potential.

2.4.4.5 Gamma-ray spectroscopy

Since bauxite residues are classified as TENORM materials, investigating their radiation levels is essential to ensure the safety of a CRM recovery project. The radiation dose can be detected using a radiation detector, such as a Geiger–Mueller tube. The Geiger tube generates a pulse of electrical current each time radiation passes through the tube and causes ionization. Each pulse is electronically detected and registers as a count (Qin & Wu, 2011). The radiation can also be measured by using spectral gamma-ray logging, which indicates the contribution of K, U, and Th to the total gamma-ray signal. The gamma-ray spectrum can be detected using portable spectrometers and scintillometers. They are increasingly being used to help calibrate airborne gamma-ray spectrometers, support geological field mapping, soil mapping, environmental applications, and monitor radioactive waste pollution (Richards, 2001).

2.4.5 Summary

A comprehensive characterization of mine wastes, particularly bauxite residue, focusing on detecting CRMs, involves mainly geochemistry and mineralogy. The geochemical characterization provides a direct approximation to the CRM content, whereas the mineralogical one gives information about the possible hosting minerals and possible mechanisms of mobility

Table 2–6 Summary of the techniques for characterization of mine wastes.

	Geochemistry	Mineralogy	Other techniques
Laboratory based	XRF LIBS INAA ICP-AES ICP-MS	Infrared spectroscopy Raman spectroscopy XRD	EMPA SEM MLA Fluid chemistry
Field based	Portable XRF Portable LIBS	Infrared spectroscopy	Mineral hardness Gamma-ray spectroscopy pH

EMPA, electron micro probe analyzer; *ICP-AES*, inductively coupled plasma atomic emission spectroscopy; *ICP-MS*, inductively coupled plasma mass spectrometry; *INAA*, instrumental neutron activation analysis; *LIBS*, laser-induced breakdown spectroscopy; *MLA*, mineral liberation analysis; *SEM*, scanning electron microscopy; *XRD*, X-ray diffraction; *XRF*, X-ray fluorescence.

and enrichment. The analysis can be performed with different levels of detail either in a laboratory environment or directly at the waste site depending upon the availability of analytical instruments. Other techniques that involve the characterization of physical properties, microscopy, and fluid chemistry support the geochemical and mineralogical analyses. Table 2−6 gives a summary of the techniques reviewed in this section.

2.5 Characterization of mining residues using remote sensing

Earth observation (EO) tools can be very useful for the preliminary mapping, quantification, and monitoring of the mining residues, usually abandoned, in harsh environments, and with limited possibilities for full sampling campaigns. Some EO sensors measure the portion of incoming solar radiation reflected by surface materials across several spectral bands (i.e., passive sensors). The physical principles that govern the data acquisition with these sensors are those of IR spectroscopy, introduced in Section 2.4. The spectral reflectance patterns for common land-cover types, such as water, bare soil, and vegetation, are well-constrained. Because of this, a remotely sensed image can be an input for classification algorithms, indicating the presence of mining, infrastructures, stockpiles, and tailings at the time of image acquisition. Some advantages of using EO concern the availability of free and easy-to-access data and the continuity of data in time with subsequent continuous land monitoring.

Outside the mining industry, academics and civil society organizations have employed EO to analyze the impacts of mining for various purposes. In this framework, most analyses have addressed mapping pollution and environmental variables in both active and abandoned mining areas, combined with imaging spectroscopy for directly identifying minerals and soils containing pollutants as an indicator of contamination. The review done by (Werner et al., 2019) is quite an exhaustive document on the use of remote sensing as a guide for sampling and environmental monitoring to demonstrate the impact of mining activities. Liang et al. (2021) provided a global-scale spatial assessment of mine areas using EO data, identifying more than 5189 tailings ponds covering approximately 1884 km^2 of the Earth's surface. Such assessment seeks to facilitate a more systematic global management of

mine wastes. All studies have proved that traditional old mining activities significantly affect the surrounding environment because of the lack of policies concerning tailings materials, leading to massive redistributions downstream from their original dumpsite to the rivers. On the other side, more recent mining activities are subjected to strict regulations, which, together with raising awareness, led to remediation and constant monitoring of closed mining sites.

2.5.1 Acquisition of earth observation data

Passive EO systems are normally classified based on their spatial and spectral resolutions. Depending on the pixel size (i.e., ground cell), the spatial resolution is subdivided into low (1 km × 1 km), medium (10 m × 10 m to 80 m × 80 m), and high (<1 m × 1 m). The spectral resolution depends on the number of bands in which the electromagnetic spectrum is captured. Passive EO systems capture the energy of the electromagnetic spectrum in the VNIR, SWIR, and LWIR (conventionally known as TIR in EO applications) ranges. Multispectral sensors record 3- to 12-wide bands (0.5−15 μm), whereas hyperspectral sensors record hundreds of narrow bands (10−20 nm). These differences in bandwidth imply that hyperspectral images contain substantially more data than multispectral ones (Limpitlaw, 2006).

The uses of EO data are also dependent on the acquisition modes. Data collected from satellite platforms and crewed/uncrewed airborne vehicles (UAVs) allow different area coverage and spatial and spectral detail levels. Images collected using satellite platforms permit surveying large extensions of the Earth's surface with periodical revisit times, which are ideal for developing monitoring tools in mine waste environments. In contrast, airborne vehicles allow for higher spatial and spectral resolution images that can be acquired in scheduled deployments and customized paths. These characteristics are favorable for specific targets, both in space and time.

2.5.1.1 Satellite (Spaceborne)

Satellite imagery has been used for several decades. Rathore and Wright (1993) reviewed the use of remote sensing to assess the impacts of coal mining and ascertained that the improvements in satellite imagery (e.g., higher resolution) would benefit the mapping of mine sites. The type and quality of information retrieved for mine waste surveys have considerably evolved since launching the Landsat missions in the 1970s. The first studies using these multispectral/medium spatial resolution images could only be used for regional surveys and the identification of different types of land use (e.g., mine waste, water body, active mine, inactive mine, and reclaimed land). Given the periodicity of the generation of EO images, those categories could be used to detect changes in time using multitemporal datasets. In those early days, image processing was not automated, and there was seldom integration with other data sources, making all the work quite demanding. Nevertheless, the results marked the path for the potential use of satellite data for detecting waste mine disposal sites (Anuta & Bahethi, 1982).

Advancements in technology and data analytics now permit to use multi- and hyperspectral space-borne sensors to map the spatial extent of mine waste surfaces and retrieve compositional information. For example, Mielke et al. (2014) mapped the 900-nm absorption characteristic of iron as a potential proxy for monitoring the extension of mine residues in South Africa. The researchers compared this feature in multispectral Landsat ETM +, Landsat-8, ASTER, and hyperspectral Hyperion images to assess the potential of the multispectral Sentinel + 2 and hyperspectral EnMAP missions. They concluded that EO data from space-borne platforms constitutes a building block of a multisensor process chain for mine waste management.

2.5.1.2 Crewed aircraft (airborne)

Crewed airborne platforms became popular in the 1990s with the launch of the airborne visible/IR imaging spectrometer (AVIRIS) instrument and became widespread in the study of mine waste sites. Airborne EO campaigns usually carry multi- and hyperspectral instruments with medium to high spatial resolution. The area coverage is determined by the flying path, typically in the scale of tens of kilometers, which is most suitable for local surveys.

One of the most used hyperspectral sensors is mounted on airborne platforms in the Australian HyMap system. An example of multitemporal studies of mine wastes concerns monitoring the Odiel River path on July 17, 2005, August 4, 2008, and August 13, 2009 (Buzzi et al., 2014). The sensor has 128 wavebands from 436 to 2485 nm with a spectral resolution of 15 nm in the 436−125 nm wavelength range, 13 nm in the 140−180 nm wavelength range, and 17 nm in the 195−248 wavelength range. Over the abandoned mining sites of the Iberian Pyrite Belt, it was possible to detect mineral changes (due to the weather) and anthropic interventions (due to remediation of mining sites) over the years. The HyMap 2005 flight pictured the state of the facilities before any recovery activity. The HyMap 2008 flight showed the ash dam covered by dry grass, completely recovered, and the surroundings clean, free from any pyrite weathering product, which was mechanically cleaned over the dumps. However, oxidation persisted due to the movement of machinery. Finally, the HyMap 2009 flight evidenced that the mud from the mill tailings dam was removed. The oxidation state in the crusts started to decrease due to the dismantling of the ore processing plant, buildings, and machinery.

2.5.1.3 Uncrewed aircraft (drone-borne)

The first reports of the deployment of lightweight UAVs equipped with spectrometric cameras date from the 2010s. In 2013 a study led by the United States Geological Survey sought to determine if UAVs could provide cost-effective, high-resolution imagery. The data retrieved by UAVs carrying optical systems and commercial cameras aided in identifying abandoned mine waste sites, determining their significance, and supporting cleanup endeavors (Cress et al., 2015). The deployment of UAVs allows the cost-effective acquisition of data with very detailed spatial resolution and small spatial coverage for environmental applications (Honkavaara et al., 2013).

Acquiring EO data with UAVs for mine waste studies has numerous advantages. UAVs improve accessibility to remote and dangerous (e.g., unstable terrains) areas that would be difficult or even impossible to survey by personnel on the ground. Systems carried by UAVs are commonly tailored to the particular needs of a project, and a single UAV can carry different sensors. Using UAVs makes it possible to perform fieldwork campaigns where remotely sensed and ground-truth data (e.g., pXRF or portable IR spectrometers) can be used simultaneously. The combination of technologies has also shortened campaign planning times (Jackisch et al., 2018; Said et al., 2021).

2.5.2 Applications in mine residues

The applications of remote sensing platforms equipped with optical systems (VNIR, SWIR, and TIR) in mining are predominant in the exploration and exploitation phases. In mine waste management, the applications focus on soil and water pollution (i.e., stream sediments, concentration of hazardous minerals and metals, ARD), and ecological restoration [i.e., distribution of vegetation (out of the scope of this chapter)] (Park & Choi, 2020). However, applications in the detection of valuable minerals and opportunities for revalorization are still developing.

2.5.2.1 Stream sediments

Various researchers have used EO to map sediments redistributed in the rivers downstream mining areas and to characterize the mineralogical variability. For example, Ferrier (1999) worked on waste rock and tailings produced from mining activities, focusing on environmental pollution of materials and trace elements of tailings using airborne mapping and high imaging spectrometer data of the Rodalquilar mining area in Spain. On the same mining site, more recently, Choe et al. (2008) used the spectral variations associated with the presence of heavy metals in stream sediments to characterize the distribution of areas affected by heavy metals Fig. 2—5. The studies showed how the dispersion of contaminated material could be obtained from imaging spectrometer data. The considered spectral absorption feature parameters showed the potential to detect heavy metals. The image-derived spectral parameters showed some efficacy in screening the streamlines affected by heavy metals to detect environmental pollution.

As a different example, Pascucci et al. (2012) used field and spectral analyses in combination with EO airborne hyperspectral data to map red mud dust waste in a bauxite tailings dam (red mud) located near Podgorica in Montenegro. The researchers identified the materials' dominant mineralogy and optical characteristics to map the red mud distribution on bare soils and river waters. The retrieved information is suitable for developing effective intervention policies and monitoring programs.

2.5.2.2 Mineral and metal contamination

Farrand and Harsanyi (1997) used the AVIRIS system to detect pollution in the Coeur d'Alene River, Idaho, taking place due to the underground silver mines near Kellogg, Idaho.

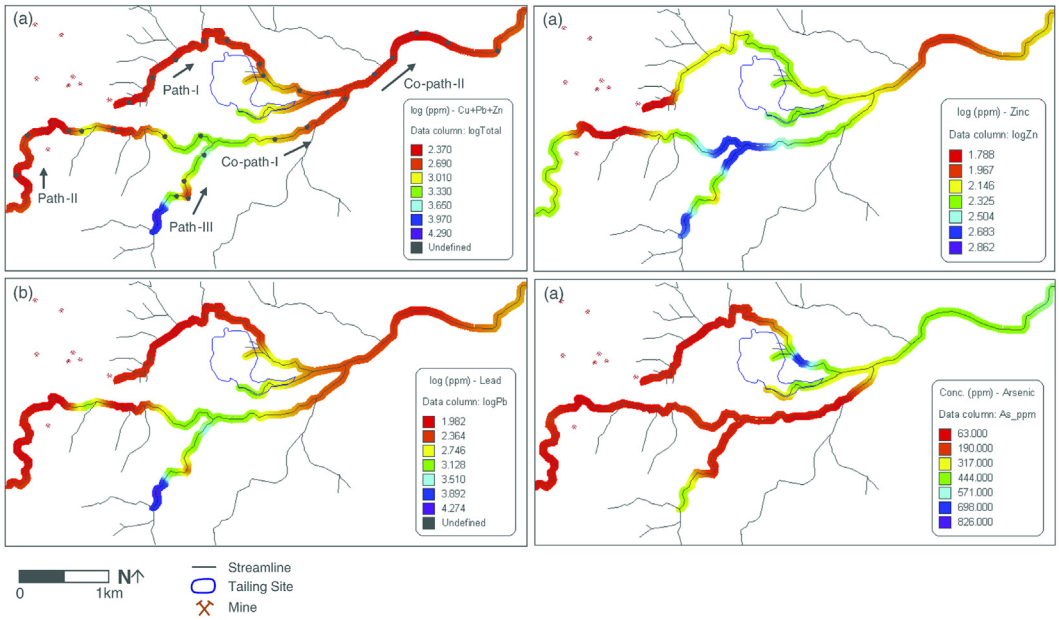

FIGURE 2–5 Streamlines and maps of concentration of heavy metals (Pb, Zn, and As) obtained by comparison between samples and hyperspectral images for the Rodalquilar mining area, Spain. *Modified from Choe, E., van der Meer, F., van Ruitenbeek, F., van der Werff, H., de Smeth, B., & Kim, K.-W. (2008). Mapping of heavy metal pollution in stream sediments using combined geochemistry, field spectroscopy, and hyperspectral remote sensing: A case study of the Rodalquilar mining area, SE Spain.* Remote Sensing of Environment*, 112*(7), *3222–3233. https://doi.org/10.1016/j.rse.2008.03.017. Reproduced with permission.*

Specifically, they used the constrained energy minimization (CEM) technique to determine the ferruginous elements of mining origin in the river (ferrihydrite). Using a specific constraint, CEM uses a finite impulse response filter to pass through the desired target while minimizing its output energy resulting from a background other than the desired targets. A correlation or covariance matrix was used to characterize the unknown composite background. A similar, more recent work, again using AVIRIS hyperspectral imagery calibrated by field observation, was done by (Mars & Crowley, 2003) over a phosphate mining zone in Idaho. In this work, metal pollutant concentrations in both vegetation and water were mapped. Specifically, eighteen mine waste dumps and five vegetation landcover types were analyzed and mapped in southeast Idaho. Relative amounts and directions of surface water flow associated with each mine dump were analyzed using digital elevation data and combined with information on stream gradients and riparian vegetation cover, providing spatial information on variations in selenium concentrations.

2.5.2.3 Acid rock drainage

Most of the EO applications in mine wastes concern the study of generators of ARD and their environmental implications, as in the example of (Swayze et al., 2000). The spectral mapping

of the Venir pile traverse wastes within the California Gulch Superfund Mining Site near Leadville (CO, USA) was obtained using AVIRIS. Specifically, the study showed how detecting jarosite at the surface has a high potential for proving the presence of acidic water. However, areas lacking jarosite at the surface may still generate acidic drainage Fig. 2−6. The authors concluded that in the case of remediated mine waste, the detection of acid mine drainage could be hindered by the presence of neutral material and vegetation. On the other hand, vegetation growth and features detectable by remote sensing can act as indicators of the presence/absence of acid drainage and pollutants.

A similar study used hyperspectral cameras deployed in a UAV to detect jarosite and goethite in the Zokolov Lignite District in the Czech Republic. Identifying the absorption bands characteristic of iron-bearing minerals formed the basis of mineral maps that were later correlated with the distribution of ground-based pH measurements. This study also showed that EO surveys improve sampling campaigns' logistics and quality. Flores et al. (2021) combined

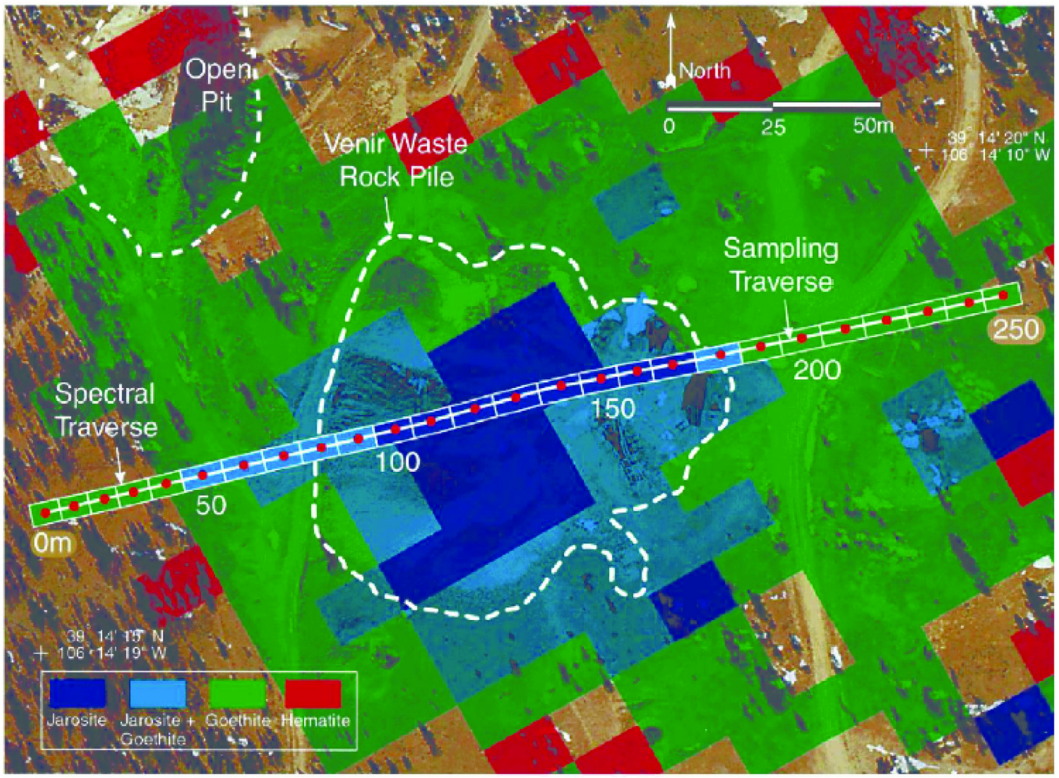

FIGURE 2–6 Spectral traverse and AVIRIS mineral maps overlaid on a high-spatial-resolution aerial photograph of the Venir traverse mine waste. *AVIRIS*, Airborne visible/infrared imaging spectrometer. *From Swayze, G. A., Smith, K. S., Clark, R. N., Sutley, S. J., Pearson, R. M., Vance, J. S., Hageman, P. L., Briggs, P. H., Meier, A. L., Singleton, M. J., & Roth, S. (2000). Using imaging spectroscopy to map acidic mine waste.* Environmental Science & Technology, 34 *(1), 47−54. https://doi.org/10.1021/es990046w.*

UAVs-acquired VNIR hyperspectral imagery with constrained hydrogeochemical data to assess the surface water quality in the Odiel-Tintillo River confluence (Spain). The authors detected gradual changes in the parameters that control water quality, particularly those related to the spatial distribution of secondary Fe minerals. In this study, the data acquisition times were faster than traditional monitoring approaches, particularly in areas of remote or dangerous access.

2.5.2.4 Revalorization of mine wastes

In recent years, the improvements in spatial and spectral resolution combined with multi-source data integration and modern data analytics have significantly leveraged the use of EO data for the revalorization of mine residues. Nowadays, it is possible to detect particular components in mine wastes and study their dynamics from space. For example, Kasmaeeyazdi, Dinelli, et al. (2022) used Sentinel-2 imagery to map the occurrence of cobalt, chromium, copper, and iron in legacy mine wastes in North Italy. The researchers surveyed the large area covered by the images to detect spots of metal enrichment that have the potential for revalorization of the mine wastes and are of interest for a more detailed investigation.

Aiming to assess the potential of recovering secondary raw materials, Kasmaeeyazdi, Braga, et al. (2022) compared Sentinel-2 multispectral images (ESA Copernicus program) with PRISMA hyperspectral images (Italian Space Agency ASI). The researchers proposed a procedure to map the concentration of metals on the surface of the residues at different scales and resolutions and evaluate the possible sources of environmental issues.

2.5.3 Summary

The characterization and monitoring of mine waste sites have greatly benefited from the deployment of EO systems. A wide range of spatial scales (i.e., space- and airborne) and spectral ranges (i.e., multi- and hyperspectral) makes EO a versatile tool. Combined with ground-truth data (i.e., analytical techniques introduced in Section 2.4), EO can retrieve information about the composition and surface distribution of mine waste materials. Identifying those patterns is critical for accurately interpreting stream sediment behavior, metal contamination estimations, and ARD predictions. Consequently, implementing similar approaches as those used for mine waste revalorization has promising potential.

2.6 Data analytics and digitalization

The characterization of mine residues using ground-, laboratory-based, and EO tools produce vast amounts of data. This situation is only likely to continue with the deployment of sensor networks and online sensors (Ghorbani et al., 2022). Transforming all that data into information and knowledge that stakeholders can ultimately use is the ultimate goal of the characterization works. The generation of models and optimized processes based on smart algorithms are the future of the raw materials sector (EIT Raw Materials, 2020). As a result,

in the last years there have been huge developments that aim to increase the efficiency in the minerals industry by using various combinations of instruments, data, and data analytics (Ghorbani et al., 2022).

Historically, data collection, management, and storage have largely depended upon the immediate goals of the company or project. However, the conditions in which data is stored can either hinder or enhance the value of the information (Kauppila et al., 2019). In recent years there has been more awareness of implementing strategies that enable the extraction of the best value from data. Such strategies comprise good practices in data acquisition, data integration, data analysis, and data sharing (Kinnunen et al., 2022). High volumes of data from varied sources, which have veracity and value, are regarded as big data (Ghorbani et al., 2022). The strategies to generate and manipulate big data and convert it into knowledge make a difference between descriptive and predictive/prescriptive data analytics (Kinnunen et al., 2022).

The European Institute of Innovation and Technology (EIT) identified six pillars for the digital transformation in the raw materials sector (EIT Raw Materials, 2020). The first two of them—artificial intelligence (AI) and machine learning (ML); virtual reality (VR) and augmented reality (AR)—largely depend upon data generated by sensors. Statistics, computational simulations, data science, and other forms of data analytics and visualization inherently make part of these pillars (Ghorbani et al., 2022).

2.6.1 Data analytics and machine learning

In the characterization of mine residues, data analytics aims to enhance the value of data by, on the one hand, integrating geochemical and mineralogical data and, on the other hand, making extra- or interpolations across different spatial scales (i.e., from a few micrometers to piles of hundreds of meters). However, thoughtful integration of datasets must be aware that all sensor technologies have limitations. Therefore to take full advantage of those technologies, there are different techniques for data fusion at pixel/point level (i.e., fusion of original data followed by classification) or feature level (i.e., extraction of discriminative features followed by fusion of extracted features) (Ghamisi et al., 2021).

Implementing ML tools offers an alternative to ground-truth data in cases where sample collection is not accessible. Machine learning tools do not focus on mineral or element abundance but create fit-for-purpose units (Ghamisi et al., 2021). In the characterization of mine residues, these units can be the same or similar to those used in the sampling strategies (Section 2.3), that is to say, geological, geometallurgical, or geoenvironmental units. In addition, some of the extracted features or units might be scale invariant. In these cases, ML algorithms can aid in mapping activities at different scales and create a smooth flow of information from the micro- to the macroscale (e.g., from hand samples to drill cores, mine faces, outcrop, and regional observations) (Thiele et al., 2021).

Integrating data of different nature (e.g., different sensor technologies, different scales) implies that the data is collected separately, imposing an additional challenge for data processing. As a solution, there is also a trend in developing single instruments that collect

different types of data (Krupnik & Khan, 2019). Nevertheless, data integration, either from the instrumentation or data analytics standpoint, is certainly the best approach to depend less on ground-truth data and process data in real-time to allow rapid decision-making and process adaptation (Ghamisi et al., 2021).

2.6.2 Virtual and augmented reality and modeling

The information and knowledge generated by ML algorithms and other data analytics are the input for more complex forms of data usage. For example, VR and AR have been proposed for monitoring mine waste deposits (Benndorf et al., 2022). Based on the data integration of multiscale and multisensor technologies, it is possible to develop AR-based platforms that characterize the dynamic processes in waste dumps. Furthermore, the integration of EO data, ground data, and AI is commercially used for applications throughout the mining value chain (EIT Raw Materials, 2020). Visualizing the information in an AR environment and producing information in real-time enables stakeholders to make better-informed decisions.

Mining residues can also be characterized by the data modeling approaches used for resource estimation. Data collected from testing and monitoring can be used as input to integrate the mineralogy of the waste deposit into geostatistical and geospatial models. In addition, the block models used typically for resource estimation can be applied to assess the potential of recovering residual ores in mine residues and the potential of generating ARD (Vaziri et al., 2022).

2.6.3 Outlook

Overall, collecting data from multiple sources at different scales and its subsequent digitalization constitutes the basis for identifying the potential of recovering valuable minerals from mine waste residues. Storing data in digital form and transforming it into useful information for stakeholders supports monitoring activities and can provide accurate knowledge of the availability, location, and quantity of recoverable minerals. Furthermore, digital platforms can enable the valorization of mine waste deposits by creating waste-to-resource matches (Kinnunen et al., 2022). A challenge remains in keeping the data accessible while still in compliance with the data protection protocols of the mining companies. Nevertheless, the availability of data and the creation of digital solutions are essential for managing and valorizing mine residues and enabling a fully integrated life-cycle approach to the entire mining value chain (European Commission, 2020).

2.7 Conclusions

This chapter reviewed the techniques, instruments, and methods suitable for mine residue sampling and characterization. We focused on the most relevant aspects for assessing the possible recovery of CRMs using the prospectivity of bauxite residue as an example. Throughout this review, we emphasized that embracing geometallurgical approaches for

sampling and characterization is significantly advantageous. From a mineral prospectivity standpoint, it was necessary to distinguish different phases of sampling. The screening phase covers large areas, has broad sampling units, and does not require high accuracy of the characterization results. The detailed sampling provides more specific information on the CRMs' content and indicators of ARD generation and wastewater and has refined material units, and the results are target specific. The strength of implementing geometallurgical concepts in the sampling phase is that it integrates parameters that are immediately aligned with the aims of the following characterization works. In this sense, the review of the characterization techniques also emphasizes those that can provide information directly at the sampling site. The advantage of using field-based characterization techniques is twofold: (1) it provides real-time information that can be used for sampling selection and optimization and (2) it gives information on the undisturbed and fresh material, which can be helpful in quality control or calibration of the laboratory techniques. Great knowledge from the mine waste dump at a larger scale can be also obtained using EO methods, which provide an overview of the mineral content and other materials. The characterization of mine residues must be an iterative process that increases in detail and accuracy while refining the specific objectives of the study at hand.

Despite the diversity of techniques and instruments, significant challenges can affect the success of a characterization campaign. For example, the lack of documentation or historical data about waste dumps can hinder the planning for the sampling works and bring considerable uncertainties. Furthermore, the outcomes of a study depend on the quality and representability of the sampled material. Achieving those two aspects can be a difficult task in environments where accessibility for workers and machines is troublesome. On the other hand, the possibility of acquiring different types of data at various scales demands high skills from researchers to make decisions. Data analytics and digitalization become then an integral part of the characterization tasks. researchers should choose the combination of techniques suitable for the type of material subjected to characterization, while providing complete information for the stated aims. In this sense, there are significant opportunities for developing a combined approach for material characterization, both at the technical level (e.g., physical combination of instruments) and in the data analysis.

References

Abzalov, M., & Abzalov, M. (2016). *Drilling techniques and drill holes logging* (pp. 39−77). Springer International Publishing. Available from https://doi.org/10.1007/978-3-319-39264-6_4.

Abzalov, M., & Newman, C. (2017). Sampling of the mineralised tailings dumps − Case study of the Mount Morgan project, central Queensland, Australia. *Applied Earth Science*, *126*(3), 124−128. Available from https://doi.org/10.1080/03717453.2017.1343927.

Anawar, H. M., Freitas, M. D. C., Canha, N., Dionísio, I., Dung, H. M., Galinha, C., & Pacheco, A. M. G. (2012). Assessment of bioaccumulation of REEs by plant species in a mining area by INAA. *Journal of Radioanalytical and Nuclear Chemistry*, *294*(3), 377−381. Available from https://doi.org/10.1007/s10967-012-1669-2.

Annels, A. E. (1991). *Mine Sampling* (pp. 43−95). Springer Science and Business Media LLC. Available from https://doi.org/10.1007/978-94-011-9714-4_2.

Anuta, M. A., & Bahethi, O. P. (1982). Mine waste location by satellite imagery. Open file report 30 May 80-18 Dec 82. PB-83-238519 Science Systems and Applications, Inc., Seabrook, MD. https://www.osti.gov/biblio/5601521

ASTM. (2007). Annual Book of ASTM standards ASTM International Unpublished content Designation D854-06, 2007, Standard test method for specific gravity of soil solids by water pycnometer 04.02.

Balaram, V. (2017). Field-portable instruments in mineral exploration: past, present and future. *Journal of Applied Geochemistry, 19*(4), 382−399.

Balaram, V. (2019). Rare earth elements: A review of applications, occurrence, exploration, analysis, recycling, and environmental impact. *Geoscience Frontiers*, *10*(4), 1285−1303. Available from https://doi.org/10.1016/j.gsf.2018.12.005; https://www.sciencedirect.com/journal/geoscience-frontiers.

Balaram, V., Anjaiah, K. V., & Reddy, M. R. P. (1995). Comparative study on the trace and rare earth element analysis of an Indian polymetallic nodule reference sample by inductively coupled plasma atomic emission spectrometry and inductively coupled plasma mass spectrometry. *The Analyst*, *120*(5), 1401−1406. Available from https://doi.org/10.1039/AN9952001401.

Benndorf, J., Restrepo, D. A., Merkel, N., John, A., Buxton, M., Guatame-Garcia, A., Dalm, M., de Waard, B., Flores, H., Möllerherm, S., Pizano Wagner, L. A., Schmidt, S., Knobloch, A., Nolte, H., & Kreßner, M. (2022). TRIM4Post-Mining: Transition information modelling for attractive post-mining landscapes—A conceptual framework. *Mining*, *2*(2), 248−277. Available from https://doi.org/10.3390/mining2020014; https://www.mdpi.com/2673-6489/2/2/14.

Bertrand, G. (2015). Compilation of mining waste databases to prioritize site study for metal recovery. Perspectives. 13th SGA Biennal Meeting.

Bhatt, C. R., Jain, J. C., Goueguel, C. L., McIntyre, D. L., & Singh, J. P. (2018). Determination of rare earth elements in geological samples using laser-induced breakdown spectroscopy (LIBS). *Applied Spectroscopy*, *72*(1), 114−121. Available from https://doi.org/10.1177/0003702817734854.

Binnemans, K., Jones, P. T., Blanpain, B., Van Gerven, T., & Pontikes, Y. (2015). Towards zero-waste valorisation of rare-earth-containing industrial process residues: A critical review. *Journal of Cleaner Production*, *99*, 17−38. Available from https://doi.org/10.1016/j.jclepro.2015.02.089; https://www.journals.elsevier.com/journal-of-cleaner-production.

Boesche, N. K., Rogass, C., Lubitz, C., Brell, M., Herrmann, S., Mielke, C., Tonn, S., Appelt, O., Altenberger, U., & Kaufmann, H. (2015). Hyperspectral REE (rare earth element) mapping of outcrops-applications for neodymium detection. *Remote Sensing*, *7*(5), 5160−5186. Available from https://doi.org/10.3390/rs70505160; http://www.mdpi.com/2072-4292/7/5/5160/pdf.

Borra, C. R., Blanpain, B., Pontikes, Y., Binnemans, K., & Van Gerven, T. (2016). Recovery of rare earths and other valuable metals from bauxite residue (red mud): A review. *Journal of Sustainable Metallurgy*, *2*(4), 365−386. Available from https://doi.org/10.1007/s40831-016-0068-2; https://link.springer.com/journal/40831.

Bodénan, F., Guézennec, A.-G., Beaulieu, M., Bellefant, G., Lemière, B., Lerouge, C., Save, M. (2015). Reprocessing of mine tailings: Discussion on case studies. 13th SGA Biennial Meeting. (2015).

Buxton, M. W. N., & Benndorf, J. (2013). The use of sensor derived data in optimization along the mine-value-chain: An overview and assessment of techno-economic significance. In *Proceedings of the 15th International ISM Congress*.

Buzzi, J., Riaza, A., García-Meléndez, E., Weide, S., & Bachmann, M. (2014). Mapping changes in a recovering mine site with hyperspectral airborne HyMap imagery (Sotiel, SW Spain). *Minerals*, *4*(2). Available from https://doi.org/10.3390/min4020313, 2075−163X.

Choe, E., van der Meer, F., van Ruitenbeek, F., van der Werff, H., de Smeth, B., & Kim, K.-W. (2008). Mapping of heavy metal pollution in stream sediments using combined geochemistry, field spectroscopy,

and hyperspectral remote sensing: A case study of the Rodalquilar mining area, SE Spain. *Remote Sensing of Environment*, *112*(7), 3222–3233. Available from https://doi.org/10.1016/j.rse.2008.03.017; https://www.sciencedirect.com/science/article/pii/S0034425708001193.

Chryssikos, G. D., & Gates, W. P. (2017). *Spectral manipulation and introduction to multivariate analysis developments in clay science* (pp. 64–106). Elsevier B.V. Available from https://doi.org/10.1016/B978-0-08-100355-8.00004-7; http://www.elsevier.com/wps/find/bookdescription.cws_home/BS_S187/description#description.

Cracknell, M. J., Parbhakar-Fox, A., Jackson, L., & Savinova, E. (2018). Automated acid rock drainage indexing from drill core imagery. *Minerals*, *8*(12). Available from https://doi.org/10.3390/min8120571; https://www.mdpi.com/2075-163X/8/12/571/pdf.

Cress, J., Hutt, M., Sloan, J., Bauer, M., Feller, M., & Goplen, S. (2015). U.S. Geological Survey U.S. Geological Survey Unmanned Aircraft Systems (UAS) Roadmap 2014.

Dalm, M., Buxton, M. W. N., & van Ruitenbeek, F. J. A. (2017). Discriminating ore and waste in a porphyry copper deposit using short-wavelength infrared (SWIR) hyperspectral imagery. *Minerals Engineering*, *105*, 10–18. Available from https://doi.org/10.1016/j.mineng.2016.12.013; http://www.journals.elsevier.com/minerals-engineering/.

Deady, E. (2014). Rare earth elements in karst-bauxites: A novel untapped European resource? In *ERES2014: 1st European rare earth resources conference*.

Deady, É. A., Mouchos, E., Goodenough, K., Williamson, B. J., & Wall, F. (2016). A review of the potential for rare-earth element resources from European red muds: Examples from Seydişehir, Turkey and Parnassus-Giona, Greece. *Mineralogical Magazine*, *80*(1), 43–61. Available from https://doi.org/10.1180/minmag.2016.080.052; http://www.ingentaconnect.com/content/minsoc/mag.

Dinwiddie, C., & Stothoff. (2013). Methods for monitoring net inflow through soil covers of uranium mill tailings impoundments.

Dominy, S. C. (2018). Geometallurgy—A Route to More Resilient Mine Operations. 8.

Dubos, J. L., Orberger, B., Milazzo, J. M., Blancher, S. B., & Lützenkirchen, J. (2018). *Physico-chemical characterisation of industrial manganese dusts and sludges and its implication for agglomeration: From ore to metal BT. 29th International Mineral Processing Congress, IMPC 2018* (pp. 3577–3587). IMPC.

Dur, J. C., Elsass, F., Chaplain, V., & Tessier, D. (2004). The relationship between particle-size distribution by laser granulometry and image analysis by transmission electron microscopy in a soil clay fraction. *European Journal of Soil Science*, *55*(2), 265–270. Available from https://doi.org/10.1111/j.1365-2389.2004.00597.x.

EIT Raw Materials. (2020).

European Commission. (2020).

Eyers, S. L. (1999). Estimating bauxite quality using FITR spectroscopy and multivariate calibration. In *Proceedings of the fifth international alumina quality workshop, 1*, 86–95.

Fagerlund, G. (1973). Determination of specific surface by the BET method. *Matériaux et Construction*, *6*(3), 239–245. Available from https://doi.org/10.1007/BF02479039.

Fahad, M., Ali, S., Shah, K. H., Shahzad, A., & Abrar, M. (2019). Quantitative elemental analysis of high silica bauxite using calibration-free laser-induced breakdown spectroscopy. *Applied Optics*, *58*(27), 7588–7596. Available from https://doi.org/10.1364/AO.58.007588; https://www.osapublishing.org/ao/abstract.cfm?uri = ao-58-27-7588.

Farmer, V. C. (1974). The infrared spectra of minerals. *Mineralogical Society of Great Britain and Ireland*. Available from https://doi.org/10.1180/mono-4.

Farrand, W. H., & Harsanyi, J. C. (1997). Mapping the distribution of mine tailings in the Coeur d'Alene River Valley, Idaho, through the use of a constrained energy minimization technique. *Remote Sensing of Environment*, *59*(1), 64–76. Available from https://doi.org/10.1016/S0034-4257(96)00080-6; https://www.sciencedirect.com/science/article/pii/S0034425796000806.

Faulstich, F. R. L., Castro, H. V., De Oliveira, L. F. C., & Neumann, R. (2011). Raman spectroscopic analysis of real samples: Brazilian bauxite mineralogy. *Spectrochimica Acta - Part A: Molecular and Biomolecular Spectroscopy*, *80*(1), 102−105. Available from https://doi.org/10.1016/j.saa.2011.03.001, 13861425.

Ferrier, G. (1999). Application of imaging spectrometer data in identifying environmental pollution caused by mining at Rodaquilar, Spain. *Remote Sensing of Environment*, *68*(2), 125−137. Available from https://doi.org/10.1016/S0034-4257(98)00105-9; https://www.sciencedirect.com/science/article/pii/S0034425798001059.

Flores, H., Lorenz, S., Jackisch, R., Tusa, L., Contreras, I. C., Zimmermann, R., & Gloaguen, R. (2021). UAS-based hyperspectral environmental monitoring of acid mine drainage affected waters. *Minerals*, *11*(2). Available from https://doi.org/10.3390/min11020182.

Fox, N., Parbhakar-Fox, A., Moltzen, J., Feig, S., Goemann, K., & Huntington, J. (2017). Applications of hyperspectral mineralogy for geoenvironmental characterisation. *Minerals Engineering*, *107*, 63−77. Available from https://doi.org/10.1016/j.mineng.2016.11.008; http://www.journals.elsevier.com/minerals-engineering/.

Gaft, M. (2005). *Luminescence spectroscopy of minerals and materials*. Springer-Verlag.

Gallhofer, D., & Lottermoser, B. (2018). The influence of spectral interferences on critical element determination with portable X-ray fluorescence (pXRF). *Minerals.*, *8*(8), 320. Available from https://doi.org/10.3390/min8080320.

GARD Guide. (2018).

Ghamisi, P., Shahi, K. R., Duan, P., Rasti, B., Lorenz, S., Booysen, R., Thiele, S., Contreras, I. C., Kirsch, M., & Gloaguen, R. (2021). The potential of machine learning for a more responsible sourcing of critical raw materials. *IEEE Journal of Selected Topics in Applied Earth Observations and Remote Sensing*, *14*, 8971−8988. Available from https://doi.org/10.1109/JSTARS.2021.3108049.

Ghorbani, Y., Zhang, S. E., Nwaila, G. T., & Bourdeau, J. E. (2022). Framework components for data-centric dry laboratories in the minerals industry: A path to science-and-technology-led innovation. *The Extractive Industries and Society*, *10*, 101089. Available from https://doi.org/10.1016/j.exis.2022.101089; https://www.sciencedirect.com/science/article/pii/S2214790X22000508.

Gorączko, A., & Topoliński, S. (2020). Particle size distribution of natural clayey soils: A discussion on the use of laser diffraction analysis (LDA). *Geosciences*, *10*(2), 2076−3263. Available from https://doi.org/10.3390/geosciences10020055.

Gräfe, M., Power, G., & Klauber, C. (2011). Bauxite residue issues: III. Alkalinity and associated chemistry. *Hydrometallurgy*, *108*(1−2), 60−79. Available from https://doi.org/10.1016/j.hydromet.2011.02.004.

Gregg, S. J., Sing, K. S. W., & Salzberg, H. W. (1967). Adsorption surface area and porosity. *Journal of the Electrochemical Society*, *114*(11), 279C. Available from https://doi.org/10.1149/1.2426447.

Gu, H., Wang, N., & Liu, S. (2012). Radiological restrictions of using red mud as building material additive. *Waste Management and Research*, *30*(9), 961−965. Available from https://doi.org/10.1177/0734242X12451308.

Guatame-Garcia, A., & Buxton, M. (2018). Prediction of soluble Al_2O_3 in calcined kaolin using infrared spectroscopy and multivariate calibration. *Minerals*, *8*(4). Available from https://doi.org/10.3390/min8040136; http://www.mdpi.com/2075-163X/8/4/136/pdf.

Guatame-Garcia, A., & Buxton, M. (2020). Framework for monitoring and control of the production of calcined kaolin. *Minerals*, *10*(5). Available from https://doi.org/10.3390/min10050403, 2075−163X.

Haavisto, O., & Hyötyniemi, H. (2011). Reflectance spectroscopy in the analysis of mineral flotation slurries. *Journal of Process Control*, *21*(2), 246−253. Available from https://doi.org/10.1016/j.jprocont.2010.10.015, 09591524.

Harmon, R. S., Russo, R. E., & Hark, R. R. (2013). Applications of laser-induced breakdown spectroscopy for geochemical and environmental analysis: A comprehensive review. *Spectrochimica Acta − Part B Atomic Spectroscopy*, *87*, 11−26. Available from https://doi.org/10.1016/j.sab.2013.05.017.

Heister, K. (2014). The measurement of the specific surface area of soils by gas and polar liquid adsorption methods—Limitations and potentials. *Geoderma*, *216*, 75−87. Available from https://doi.org/10.1016/j.geoderma.2013.10.015; https://www.sciencedirect.com/science/article/pii/S0016706113003686.

Heuer, M., & Leschonski, K. (1985). Results obtained with a new instrument for the measurement of particle size distributions from diffraction patterns. *Particle & Particle Systems Characterization*, *2*(1-4), 7−13. Available from https://doi.org/10.1002/ppsc.19850020102.

Honkavaara, E., Saari, H., Kaivosoja, J., Pölönen, I., Hakala, T., Litkey, P., Mäkynen, J., & Pesonen, L. (2013). Processing and assessment of spectrometric, stereoscopic imagery collected using a lightweight UAV spectral camera for precision agriculture. *Remote Sensing*, *5*(10), 5006−5039. Available from https://doi.org/10.3390/rs5105006; https://www.mdpi.com/2072-4292/5/10/5006.

Hubbard. (2020).

Hunt, G. R. (1977). Spectral signatures of particulate minerals in the visible and near infrared. *Geophysics.*, *42*(3), 501−513. Available from https://doi.org/10.1190/1.1440721.

Ibrahim, E., Barnabé, P., Ramanaidou, E., & Pirard, E. (2018). Mapping mineral chemistry of a lateritic outcrop in new Caledonia through generalized regression using Sentinel-2 and field reflectance spectra. *International Journal of Applied Earth Observation and Geoinformation*, *73*, 653−665. Available from https://doi.org/10.1016/j.jag.2018.08.004; http://www.elsevier.com/locate/jag.

Jackisch, R., Lorenz, S., Zimmermann, R., Möckel, R., & Gloaguen, R. (2018). Drone-borne hyperspectral monitoring of acid mine drainage: An example from the Sokolov Lignite district. *Remote Sensing*, *10*(3). Available from https://doi.org/10.3390/rs10030385.

Jamieson, H. E. (2011). Geochemistry and mineralogy of solid mine waste: Essential knowledge for predicting environmental impact. *Elements*, *7*(6), 381−386. Available from https://doi.org/10.2113/gselements.7.6.381; http://elementsmagazine.org/processIP.lasso?number = e7_6&filename = e7_6.pdf.

Jamieson, H. E., Walker, S. R., & Parsons, M. B. (2015). Mineralogical characterization of mine waste. *Applied Geochemistry*, *57*, 85−105. Available from https://doi.org/10.1016/j.apgeochem.2014.12.014; http://www.journals.elsevier.com/applied-geochemistry.

Jiangang, F., Kaida, C., Hui, W., Chao, G., & Wei, L. (2012). Recovering molybdenite from ultrafine waste tailings by oil agglomerate flotation. *Minerals Engineering*, *39*, 133−139. Available from https://doi.org/10.1016/j.mineng.2012.07.006; https://www.sciencedirect.com/science/article/pii/S0892687512002579.

Kasmaeeyazdi, S., Braga, R., Tinti, F., & Mandanici, E. (2022). Mapping bauxite mining residues using remote sensing techniques. *Materials Proceedings*, *5*(1), 91. Available from: https://doi.org/10.3390/materproc2021005091; https://www.mdpi.com/2673-4605/5/1/91.

Kasmaeeyazdi, S., Dinelli, E., & Braga, R. (2022). Mapping Co−Cr−Cu and Fe Occurrence in a legacy mining waste using geochemistry and satellite imagery analyses. *Applied Sciences*, *12*(4), 1928. Available from https://doi.org/10.3390/app12041928; https://www.mdpi.com/2076-3417/12/4/1928.

Kauppila, T., Bellenfant, G., Solismaa, L., Mittelstadt, P., Fourie, A. B., & Tibbett, M. (2019). *Digitalisation of continuous mine closure planning and management: An EIT RawMaterials initiative Digitalisation of continuous mine closure planning and management* (pp. 1023−1030). Australian Centre for Geomechanics. https://papers.acg.uwa.edu.au/p/1915_81_Kauppila/.

Keeney, L., & Nguyen, K. (2014). The use of EQUOtip as a hardness domaining tool. *International Mineral Processing Congress*, *1*, 1412−1413.

King, P. L., Ramsey, M. S., McMillan, P. F., & Swayze, G. A. (2004). Laboratory Fourier transform infrared spectroscopy methods for geologic samples Short Courses Series. *Mineralogical Association of Canada*, *33*, 57−92.

Kinnunen, P., Karhu, M., Yli-Rantala, E., Kivikytö-Reponen, P., & Mäkinen, J. (2022). A review of circular economy strategies for mine tailings. *Cleaner Engineering and Technology*, *8*100499. Available from https://doi.org/10.1016/j.clet.2022.100499; https://www.sciencedirect.com/science/article/pii/S2666790822001045.

Kirsch, M., Lorenz, S., Zimmermann, R., Tusa, L., Möckel, R., Hödl, P., Booysen, R., Khodadadzadeh, M., & Gloaguen, R. (2018). Integration of terrestrial and drone-borne hyperspectral and photogrammetric sensing methods for exploration mapping and mining monitoring. *Remote Sensing*, *10*(9), 1366. Available from https://doi.org/10.3390/rs10091366.

Klauber, C., Gräfe, M., & Power, G. (2011). Bauxite residue issues: II. Options for residue utilization. *Hydrometallurgy*, *108*(1-2), 11−32. Available from https://doi.org/10.1016/j.hydromet.2011.02.007.

Knobloch, M., & Lottermoser, B. G. (2020). Infrared thermography: A method to visualise and analyse sulphide oxidation. *Minerals*, *10*(11), 1−21. Available from https://doi.org/10.3390/min10110933; https://www.mdpi.com/2075-163X/10/11/933/pdf.

Krupnik, D., & Khan, S. (2019). Close-range, ground-based hyperspectral imaging for mining applications at various scales: Review and case studies. *Earth-Science Reviews*, *198*, 102952. Available from https://doi.org/10.1016/j.earscirev.2019.102952; https://www.sciencedirect.com/science/article/pii/S001282521930251X.

Kuhn, K., Meima, J. A., Rammlmair, D., & Ohlendorf, C. (2016). Chemical mapping of mine waste drill cores with laser-induced breakdown spectroscopy (LIBS) and energy dispersive X-ray fluorescence (EDXRF) for mineral resource exploration. *Journal of Geochemical Exploration*, *161*, 72−84. Available from https://doi.org/10.1016/j.gexplo.2015.11.005.

Laakso, K., Turner, D. J., Rivard, B., & Sánchez-Azofeifa, A. (2019). The long-wave infrared (8-12 μm) spectral features of selected rare earth element—Bearing carbonate, phosphate and silicate minerals. *International Journal of Applied Earth Observation and Geoinformation*, *76*, 77−83. Available from https://doi.org/10.1016/j.jag.2018.11.005; http://www.elsevier.com/locate/jag.

Lapakko, K. (2002). *Metal mine rock and waste characterization tools: An overview*. Mining, Minerals and Sustainable Development is a project of the International Institute for Environment and Development.

Laser distance spectrometry. (2017).

Latimer, C. (2010). Managing heads and tails. *Australian Mining*, *102*(4), 14−16.

Lawley, C. J. M., Somers, A. M., & Kjarsgaard, B. A. (2021). Rapid geochemical imaging of rocks and minerals with handheld laser induced breakdown spectroscopy (LIBS). *Journal of Geochemical Exploration*, 222. Available from https://doi.org/10.1016/j.gexplo.2020.106694; http://www.sciencedirect.com/science/journal/03756742.

Lemière, B. (2018). A review of pXRF (field portable X-ray fluorescence) applications for applied geochemistry. *Journal of Geochemical Exploration*, *188*, 350−363. Available from https://doi.org/10.1016/j.gexplo.2018.02.006; http://www.sciencedirect.com/science/journal/03756742.

Lenz, C., Nasdala, L., Talla, D., Hauzenberger, C., Seitz, R., & Kolitsch, U. (2015). Laser-induced REE3 + photoluminescence of selected accessory minerals − An \advantageous artefact\ in Raman spectroscopy. *Chemical Geology*, *415*, 1−16. Available from https://doi.org/10.1016/j.chemgeo.2015.09.001; http://www.sciencedirect.com/science/journal/00092541.

Liang, T., Werner, T. T., Heping, X., Jingsong, Y., & Zeming, S. (2021). A global-scale spatial assessment and geodatabase of mine areas. *Global and Planetary Change*, *204*, 103578. Available from https://doi.org/10.1016/j.gloplacha.2021.103578; https://www.sciencedirect.com/science/article/pii/S0921818121001636.

Limpitlaw, D. (2006). Use of remotely sensed imagery and methods for mapping and planning of mine waste facilities. 9.

Liu, Y., Lin, C., & Wu, Y. (2007). Characterization of red mud derived from a combined Bayer Process and bauxite calcination method. *Journal of Hazardous Materials*, *146*(1−2), 255−261. Available from https://doi.org/10.1016/j.jhazmat.2006.12.015.

Madejová, J., Balan, E., & Petit, S. (2011). Application of vibrational spectroscopy to the characterization of phyllosilicates and other industrial minerals. 9. https://doi.org/10.1180/emu-notes.2010.emu9-6.

Malvern Panalytical. (2021).

Marion, R., & Carrère, V. (2018). Mineral mapping using the Automatized Gaussian Model (AGM)-application to two industrial French sites at Gardanne and Thann. *Remote Sensing*, *10*(1). Available from https://doi.org/10.3390/rs10010146; http://www.mdpi.com/2072-4292/10/1/146/pdf.

Marjoribanks, R. (2010). *Drilling: A general discussion the importance of drilling. Geological methods in mineral exploration and mining* (pp. 75–84). Springer Berlin Heidelberg. Available from https://doi.org/10.1007/978-3-540-74375-0_5.

Mars, J. C., & Crowley, J. K. (2003). Mapping mine wastes and analyzing areas affected by selenium-rich water runoff in southeast Idaho using AVIRIS imagery and digital elevation data. *Remote Sensing of Environment*, *84*(3), 422–436. Available from https://doi.org/10.1016/S0034-4257(02)00132-3; https://www.sciencedirect.com/science/article/pii/S0034425702001323.

McGuiness, L. (2005). Automated analysis of bauxite exploration drill hole samples by diffuse reflectance – Fourier Transform Infrared (FTIR) spectroscopy. In: *Seventh international alumina quality workshop* (pp. 187–192).

Mielke, C., Boesche, N. K., Rogass, C., Kaufmann, H., Gauert, C., & de Wit, M. (2014). Spaceborne mine waste mineralogy monitoring in South Africa, applications for modern push-broom missions: Hyperion/OLI and EnMAP/Sentinel-2. *Remote Sensing*, *6*(8), 6790–6816. Available from https://doi.org/10.3390/rs6086790; http://www.mdpi.com/2072-4292/6/8/6790/pdf.

Morin, K. A., & Hutt, N. M. (1995). Kinetic test and risk assessment for ARD. In: *5th annual BC metal leaching and ARD workshop*. Vancouver.

Muir, A., Mitchell, J., Flatman, S., & Sabbagha, C. (2016). *Retreatment of gold residues* (pp. 709–728). Elsevier BV. Available from https://doi.org/10.1016/b978-0-444-63658-4.00039-6.

Müller, S., Meima, J. A., & Rammlmair, D. (2021). Detecting REE-rich areas in heterogeneous drill cores from Storkwitz using LIBS and a combination of k-means clustering and spatial raster analysis. *Journal of Geochemical Exploration*, *221*, 106697. Available from https://doi.org/10.1016/j.gexplo.2020.106697.

Nikraz, H. R., Bodley, A. J., Cooling, D. J., Kong, P. Y., & Soomro, M. (2007). Comparison of physical properties between treated and untreated bauxite residue mud. *Journal of Materials in Civil Engineering*, *19*(1), 2–9. Available from https://doi.org/10.1061/(ASCE)0899-1561(2007)19:1(2).

Nordstrom, D. K. (2011). Mine waters: Acidic to circmneutral. *Elements*, *7*(6), 393–398. Available from https://doi.org/10.2113/gselements.7.6.393.

Ochsenkühn-Petropulu, M., Lyberopulu, T., & Parissakis, G. (1994). Direct determination of landthanides, yttrium and scandium in bauxites and red mud from alumina production. *Analytica Chimica Acta*, *296*(3), 305–313. Available from https://doi.org/10.1016/0003-2670(94)80250-5.

O'connor, B. H., Donoghue, A. M., Manning, T. J. H., & Chesson, B. J. (2013). Radiological assessment for bauxite mining and alumina refining. *Annals of Occupational Hygiene*, *57*(1), 63–76. Available from https://doi.org/10.1093/annhyg/mes052.

Ortiz, J. M., & Magri, E. J. (2014). Designing an advanced RC drilling grid for short-term planning in open pit mines: Three case studies. *Journal of the Southern African Institute of Mining and Metallurgy*, *114*(8), 631–637. http://www.saimm.co.za/Journal/v114n08p631.pdf.

Pan, H., Zhou, G., Cheng, Z., Yang, R., He, L., Zeng, D., & Sun, B. (2014). Advances in geochemical survey of mine tailings project in China. *Journal of Geochemical Exploration*, *139*, 193–200. Available from https://doi.org/10.1016/j.gexplo.2013.07.012.

Parbhakar-Fox, A. (2017). *Predicting waste properties using the geochemistry-mineralogy-texture-geometallurgy approach* (pp. 73–96). Springer International Publishing. https://doi.org/10.1007/978-3-319-42731-7_5.10.1007/978-3-319-42731-7_5.

Parbhakar-Fox, A., Glen, J., & Raimondo, B. (2018). A geometallurgical approach to tailings management: An example from the Savage River Fe-Ore Mine, Western Tasmania. *Minerals.*, *8*(10), 454. Available from https://doi.org/10.3390/min8100454.

Parbhakar-Fox, A., & Lottermoser, B. G. (2015). A critical review of acid rock drainage prediction methods and practices. *SI: Process Mineralogy*, *82*, 107−124. Available from https://doi.org/10.1016/j.mineng.2015.03.015; https://www.sciencedirect.com/science/article/pii/S0892687515001053.

Park, S., & Choi, Y. (2020). Applications of unmanned aerial vehicles in mining from exploration to reclamation: A review. *Minerals*, *10*(8), 663. Available from https://doi.org/10.3390/min10080663; https://www.mdpi.com/2075-163X/10/8/663.

Pascucci, S., Belviso, C., Cavalli, R. M., Palombo, A., Pignatti, S., & Santini, F. (2012). Using imaging spectroscopy to map red mud dust waste: The Podgorica Aluminum Complex case study. *Remote Sensing of Environment*, *123*, 139−154. Available from https://doi.org/10.1016/j.rse.2012.03.017; https://www.sciencedirect.com/science/article/pii/S0034425712001368.

Power, G., Gräfe, M., & Klauber, C. (2011). Bauxite residue issues: I. Current management, disposal and storage practices. *Hydrometallurgy*, *108*(1-2), 33−45. Available from https://doi.org/10.1016/j.hydromet.2011.02.006.

Prakash, K., Sridharan, A., Thejas, H. K., & Swaroop, H. M. (2012). A simplified approach of determining the specific gravity of soil solids. *Geotechnical and Geological Engineering*, *30*(4), 1063−1067. Available from https://doi.org/10.1007/s10706-012-9521-6.

Qin, S., & Wu, B. (2011). Reducing the radiation dose of red mud to environmentally acceptable levels as an example of novel ceramic materials. *Green Chemistry*, *13*(9), 2423−2427. Available from https://doi.org/10.1039/c1gc15452d.

Ramanaidou, E., Wells, M., Lau, I., & Laukamp, C. (2015). *Characterization of iron ore by visible and infrared reflectance and, Raman spectroscopiesIron Ore: Mineralogy, Processing and Environmental Sustainability* (pp. 191−228). Elsevier Inc. Available from https://doi.org/10.1016/B978-1-78242-156-6.00006-X; http://www.sciencedirect.com/science/book/9781782421566.

Rathore, C. S., & Wright, R. (1993). Monitoring environmental impacts of surface coal mining. *International Journal of Remote Sensing*, *14*(6), 1021−1042. Available from https://doi.org/10.1080/01431169308904394.

Richards, D. (2001). Calibration and use of portable gamma-ray spectrometers. *Preview − Australian Society of Exploration Geophysicists* (95), 21−23.

Rietveld, H. M. (1969). A profile refinement method for nuclear and magnetic structures. *Journal of Applied Crystallography*, *2*(2), 65−71. Available from https://doi.org/10.1107/s0021889869006558.

Rost, E., Hecker, C., Schodlok, M., & van der Meer, F. (2018). Rock sample surface preparation influences thermal infrared spectra. *Minerals.*, *8*(11), 475. Available from https://doi.org/10.3390/min8110475.

Price, W. A. (2009). Prediction manual for drainage chemistry from sulphidic geologic materials. 12. Natural Resources Canada. http://mend-nedem.org/wp-content/uploads/1.20.1_PredictionManual.pdf.

Ruan, H. D. (2005). *Spectroscopic studies of nano-structures of Al and Fe phases, bauxite and their thermally activated products*. Centre for Instrumental and Developmental Chemistry.

Ruan, H. D., Frost, R. L., & Kloprogge, J. T. (2001). Comparison of Raman spectra in characterizing gibbsite, bayerite, diaspore and boehmite. *Journal of Raman Spectroscopy*, *32*(9), 745−750. Available from https://doi.org/10.1002/jrs.736.

Sädbom, S., & Bäckström, M. (2018). Sampling of mining waste − Historical background, experiences and suggested methods.

Said, K. O., Onifade, M., Githiria, J. M., Abdulsalam, J., Bodunrin, M. O., Genc, B., Johnson, O., & Akande, J. M. (2021). On the application of drones: a progress report in mining operations. *International Journal of Mining, Reclamation and Environment*, *35*(4), 235−267. Available from https://doi.org/10.1080/17480930.2020.1804653.

Salisbury, J. W., & Wald, A. (1992). The role of volume scattering in reducing spectral contrast of reststrahlen bands in spectra of powdered minerals. *Icarus*, *96*(1), 121−128. Available from https://doi.org/10.1016/0019-1035(92)90009-V.

Santini, T. C. (2015). Application of the Rietveld refinement method for quantification of mineral concentrations in bauxite residues (alumina refining tailings). *International Journal of Mineral Processing*, *139*, 1−10. Available from https://doi.org/10.1016/j.minpro.2015.04.004; http://www.elsevier.com/inca/publications/store/5/0/3/3/3/8/.

Schodlok, M. C., Whitbourn, L., Huntington, J., Mason, P., Green, A., Berman, M., Coward, D., Connor, P., Wright, W., Jolivet, M., & Martinez, R. (2016). HyLogger-3, a visible to shortwave and thermal infrared reflectance spectrometer system for drill core logging: Functional description. *Australian Journal of Earth Sciences*, *63*(8), 929−940. Available from https://doi.org/10.1080/08120099.2016.1231133; http://www.univie.ac.at/ajes.

Sciaps. (2017).

Secopta. (2017).

Smith, K. S. (2000). Sampling strategy for the rapid screening of mine-waste dumps on abandoned mine lands. In *Proceedings, fifth international conference on acid rock drainage*.

Sonicsampdrill. (2017).

Swayze, G. A., Smith, K. S., Clark, R. N., Sutley, S. J., Pearson, R. M., Vance, J. S., Hageman, P. L., Briggs, P. H., Meier, A. L., Singleton, M. J., & Roth, S. (2000). Using imaging spectroscopy to map acidic mine waste. *Environmental Science & Technology*, *34*(1), 47−54. Available from https://doi.org/10.1021/es990046w.

Terzano, R., Denecke, M. A., Falkenberg, G., Miller, B., Paterson, D., & Janssens, K. (2019). Recent advances in analysis of trace elements in environmental samples by X-ray based techniques (IUPAC Technical Report). *Pure and Applied Chemistry*, *91*(6), 1029−1063. Available from https://doi.org/10.1515/pac-2018-0605; http://www.degruyter.com/view/j/pac.

ThermoFisher. (2017a).

ThermoFisher. (2017b).

Thiele, S. T., Lorenz, S., Kirsch, M., Cecilia Contreras Acosta, I., Tusa, L., Herrmann, E., Möckel, R., & Gloaguen, R. (2021). Multi-scale, multi-sensor data integration for automated 3-D geological mapping. *Ore Geology Reviews*, *136*, 104252. Available from https://doi.org/10.1016/j.oregeorev.2021.104252; https://www.sciencedirect.com/science/article/pii/S016913682100278X.

Thompson, A. J. B., Hauff, P. L., & Robitaille, A. J. (1999). Alteration mapping in exploration: Application of short-wave infrared (SWIR) spectroscopy. *SEG Discovery* (39), 1−27. Available from https://doi.org/10.5382/segnews.1999-39.fea.

Turner, D. J., Rivard, B., & Groat, L. A. (2014). Visible and short-wave infrared reflectance spectroscopy of REE fluorocarbonates. *American Mineralogist*, *99*(7), 1335−1346. Available from https://doi.org/10.2138/am.2014.4674; https://www.degruyter.com/view/j/ammin.

Turner, D. J., Rivard, B., & Groat, L. A. (2016). Visible and short-wave infrared reflectance spectroscopy of REE phosphate minerals. *American Mineralogist*, *101*(10), 2264−2278. Available from https://doi.org/10.2138/am-2016-5692; https://www.degruyter.com/view/j/ammin.

Ujaczki, É., Feigl, V., Molnár, M., Cusack, P., Curtin, T., Courtney, R., O'Donoghue, L., Davris, P., Hugi, C., Evangelou, M. W. H., Balomenos, E., & Lenz, M. (2018). Re-using bauxite residues: Benefits beyond (critical raw) material recovery. *Journal of Chemical Technology and Biotechnology*, *93*(9), 2498−2510. Available from https://doi.org/10.1002/jctb.5687; http://onlinelibrary.wiley.com/journal/10.1002/(ISSN)1097-4660.

van Ruitenbeek, F. J. A., van der Werff, H. M. A., Bakker, W. H., van der Meer, F. D., & Hein, K. A. A. (2019). Measuring rock microstructure in hyperspectral mineral maps. *Remote Sensing of Environment*, *220*, 94−109. Available from https://doi.org/10.1016/j.rse.2018.10.030; http://www.elsevier.com/inca/publications/store/5/0/5/7/3/3.

Vaziri, V., Sayadi, A. R., Parbhakar-Fox, A., Mousavi, A., & Monjezi, M. (2022). Improved mine waste dump planning through integration of geochemical and mineralogical data and mixed integer programming:

Reducing acid rock generation from mine waste. *Journal of Environmental Management*, *309*, 114712. Available from https://doi.org/10.1016/j.jenvman.2022.114712; https://www.sciencedirect.com/science/article/pii/S0301479722002857.

Vind, J., Malfliet, A., Blanpain, B., Tsakiridis, P., Tkaczyk, A., Vassiliadou, V., & Panias, D. (2018). Rare earth element phases in bauxite residue. *Minerals*, *8*(2), 77. Available from https://doi.org/10.3390/min8020077.

Vind, J., Malfliet, A., Bonomi, C., Paiste, P., Sajó, I. E., Blanpain, B., Tkaczyk, A. H., Vassiliadou, V., & Panias, D. (2018). Modes of occurrences of scandium in Greek bauxite and bauxite residue. *Minerals Engineering*, *123*, 35−48. Available from https://doi.org/10.1016/j.mineng.2018.04.025; http://www.journals.elsevier.com/minerals-engineering/.

Vukotić, P. (1983). Determination of rare earth elements in bauxites by instrumental neutron activation analysis. *Journal of Radioanalytical Chemistry*, *78*(1), 105−115. Available from https://doi.org/10.1007/BF02519755.

Wells, M. A., & Ramanaidou, E. R. (2015). *Raman spectroscopic core scanning for iron ore and BIF characterization* (pp. 387−396). Springer Science and Business Media LLC. Available from https://doi.org/10.1007/978-3-319-13948-7_39.

Werner, T. T., Bebbington, A., & Gregory, G. (2019). Assessing impacts of mining: Recent contributions from GIS and remote sensing. *The Extractive Industries and Society*, *6*(3), 993−1012. Available from https://doi.org/10.1016/j.exis.2019.06.011; https://www.sciencedirect.com/science/article/pii/S2214790X19300516.

Yaroshchyk, P., Death, D. L., & Spencer, S. J. (2010). Quantitative measurements of loss on ignition in iron ore using laser-induced breakdown spectroscopy and partial least squares regression analysis. *Applied Spectroscopy*, *64*(12), 1335−1341. Available from https://doi.org/10.1366/000370210793561600000037028.