





# Mine Waste



Mining, Processing and Smelter Dumps – Handbook for Exploration and Recovery Methods for Secondary Raw Material Deposits

## Imprint

Editors:	Philipp Büttner, Helmholtz Institute Freiberg for Resource Technology (HIF) at the Helmholtz-Zentrum Dresden-Rossendorf (HZDR)			
	Dr. Jochen Nühlen, Fraunhofer Institute for Environmental, Safety and Energy Technology UMSICHT Oberhausen			
	Kerstin Kuhn, Federal Institute for Geosciences and Natural Resources (BGR) (Bundesanstalt für Geowissenschaften und Rohstoffe)			
Co-Authors:	Christian Poggendorf, Prof. Burmeier Ingenieurgesellschaft mbH Dr. Asja Mrotzek-Blöß, former Fraunhofer Institute for Environmental, Safety and Energy Technology UMSICHT, now Clausthal University of Technology Michael Jandewerth, former Fraunhofer Institute for Environmental, Safety and Energy Technology UMSICHT, now Open Grid Europe GmbH Dr. Michael Denk, Martin-Luther-Universität Halle-Wittenberg Prof. Cornelia Gläßer, Martin-Luther-Universität Halle-Wittenberg Sebastian Teuwsen, EFTAS Fernerkundung und Technologietransfer GmbH Adrian Klink, EFTAS Fernerkundung und Technologietransfer GmbH David Algermissen, FEhS-Institut für Baustoff-Forschung e.V. Dr. Inga Osbahr, former Helmholtz Institute Freiberg for Resource Technology (HIF), now: Federal Institute for Geosciences and Natural Resources (BGR) Rene Luhmer, TU Bergakademie Freiberg Dr. Carsten Pätzold, TU Bergakademie Freiberg Dr. Carsten Pätzold, TU Bergakademie Freiberg Dr. Thomas Leißner, TU Bergakademie Freiberg Dr. Thomas Leißner, TU Bergakademie Freiberg Ulrich Peuser, AKW Apparate + Verfahren GmbH Mirko Martin, G.E.O.S. Freiberg Ingenieurgesellschaft mbH Dr. Tina Martin, former Federal Institute for Geosciences and Natural Resources (BGR), now: Lund University Dr. Rudolf Knieß, former Federal Institute for Geosciences and Natural Resources (BGR), now: Lund University Dr. Rudolf Knieß, former Federal Institute for Geosciences and Natural Resources (BGR) Dr. Jeannet Meima, Federal Institute for Geosciences and Natural Resources (BGR) Dr. Jeannet Meima, Federal Institute for Geosciences and Natural Resources (BGR) Dr. Jeannet Meima, Federal Institute for Geosciences and Natural Resources (BGR) Dr. Dieter RammImair, Federal Institute for Geosciences and Natural Resources (BGR) Dr. Dieter RammImair, Federal Institute for Geosciences and Natural Resources (BGR) Dr. Dieter RammImair, Federal Institute for Geosciences and Natural Resources (BGR) Dr. Dieter RammImair, Federal Institute for Geosciences and Natural Resources (BGR) Dr. Dieter RammImair, Federal			
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## Mining, Processing and Smelter Dumps – Handbook for Exploration and Recovery Methods for Secondary Raw Material Deposits

Edited by P. Büttner, J. Nühlen and K. Kuhn

January 2022

#### Preface

The German Federal Ministry of Education and Research (BMBF) has set up a funding program in 2012, named "r<sup>3</sup>- Innovative Technologien für Ressourceneffizienz – Strategische Metalle und Minerale" which dealt with the development of new technologies for urban mining and substitution or recycling of strategic elements such as indium, germanium or the rare earth elements. They are defined by the European Union as critical and their availability has a strategic importance for the German high tech industry. Within this funding program there were three projects dealing with the recovery of metals from different mine waste dumps containing residues of mining, ore processing and metal smelting. These projects have joined to form the German r<sup>3</sup> mine waste cluster to share their experiences in exploration, sampling, processing and metal recovery for dumps and furthermore to combine selected data in a German cadaster for mine waste dumps. The cadaster is now under supervision of the Institute for Geosciences and Natural Resources Federal (Bundesanstalt für Geowissenschaften und Rohstoffe, BGR) in Hannover and is expected to be published online in 2022. This handbook combines specific methods for the interdisciplinary research on residues from mining, processing and smelting that were used in the three research projects. These methods show some of the procedures that can be used for exploration, sampling, processing, metal recovery, and reclamation of mine waste dumps. The handbook has been compiled by the editors, the authors of each method are presented within the method sheet itself. The editors point out that the handbook provides an spotlight with status as of 2016, if not marked otherwise, and does not claim to be complete regarding urban mining technologies. It should therefore be seen as an inspiration for researchers and companies in this field and as an incentive for the exchange and continuation of this important topic in the course of resource and environmental protection.

The r<sup>3</sup> mine waste cluster included the following three research projects:

<u>SMSB</u> – Gewinnung strategischer Metalle und Minerale aus sächsischen Bergbauhalden (Strategic Metals and Minerals from Saxonian Mining Dumps, FKZ: 033R095)

*Contact: Philipp Büttner, Helmholtz Institute Freiberg for Resource Technology* (*p.buettner@hzdr.de, Tel.:* +49-(0)351-260-4417)

<u>ReStrateGIS</u> – Konzeption und Entwicklung eines Ressourcenkatasters für Hüttenhalden durch Einsatz von Geoinformationstechnologien und Strategieentwicklung zur Wiedergewinnung von Wertstoffen (Conception and development of resource land register for smelter heaps applying geoinformation technologies and new strategies for recycling resources, FKZ: 033R103)

Contact: Jochen Nühlen, Fraunhofer Institute for Environmental, Safety and Energy Technology UMSICHT Oberhausen (jochen.nuehlen@umsicht.fraunhofer.de, Tel.: +49-(0)208-8598-1370)

<u>ROBEHA</u> – Nutzung des Rohstoffpotenzials von Bergbau- und Hüttenhalden unter Berücksichtigung der Nachhaltigkeit am Beispiel des Westharzes (Economic potential of mine waste dumps of the Western Harz Mountains under consideration of sustainability; FKZ: 033R105) Contact: Christian Poggendorf, Prof. Burmeier Ingenieurgesellschaft mbH (c.poggendorf@burmeier-ingenieure.de, Tel.: +49-(0)511-8992230) or Kerstin Kuhn, Federal Institute for Geosciences and Natural Resources, BGR (kerstin.kuhn@bgr.de, Tel.: +49-(0)511-643-3370)

All three research projects have already been completed. Further follow-up projects in Germany that deal with the recovery of raw materials from mine waste dumps include, among others:

<u>recomine</u> – resource-oriented environmental technologies for the 21st century

(2018 – 2025, WIR! funding program, FKZ 03WIR1902)



**<u>REMINTA</u>** – Utilisation of mineral materials from the Bollrich tailings ponds (Goslar/Germany)

(2021 – 2024, ReMin funding program, FKZ 033R266H)



The recomine-alliance (www.recomine.net) is a followup project of the BMBF r<sup>3</sup> project SMSB and bundles regional, Saxonian know-how in the field of environmental technologies for combining remediation of contaminated sites (e.g. the recomine testing and demonstration sites in the Saxonian Ore Mountains) with recovery of raw materials. Topic issues of the recomine-projects include (1) Tailings and Mining Dumps, (2) Mine and Tailing Drainage Waters as well as (3) Slags and Ashes.

After the REWITA project (2015 – 2018) has already investigated the recovery of metals from tailings from the Rammelsberg deposit, the follow-up project REMINTA (www.reminta.de) aims to make the previously unusable mineral residues from metal recovery usable as well. Possible applications for these mineral residues would be in the cement industry and partly in seal construction as a substitute for power plant ash, which is becoming less abundant due to the energy transition. The aim is to develop an overall recycling concept for the Bollrich tailings ponds.

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#### 1 Detection and Modelling of Mine Waste Dumps

#### 1.1 Localization of Dumps

#### 1.1.1 Reference Database

#### Author: Jochen Nühlen

#### **Objectives:**

Creation of a database for the future utilization of secondary raw materials in mine dumps on the basis of various data. These may include both geospatial and technical data from public (official) institutions and the private sector. The particular challenge thereby is the compilation and harmonization of secondary and primary data of different quality and spatial resolution from relevant public organizations (official data) and from private, commercial information sources for the validation and detection of anthropogenic deposits.

#### Method description:

In order to generate a basis for the detailed analysis of one or more prospective anthropogenic deposits in an unknown area, various data sources (primary and secondary data) have to be collected and harmonized. Therefore, data both from public bodies and the private sector have to be captured and entered. This will serve as a basis for detecting first regions for detailed analyses, which have to be worked out in more depth by further inquiries to subordinate regional authorities and municipalities. Data from public bodies will be completed by records from private companies that also manage or produce geospatial data. In a further step, the characteristics of the mine dumps are entered and managed in tables as alphanumeric attributes (technical data).

#### Field of Application:

Application field and use: Preliminary analysis for the detection and characterization of mine dumps in unknown areas.

#### **Operational Conditions and Limitations:**

Most data from public authorities and private enterprises (e.g. agricultural associations) are not free of charge and not accessible to the public, but maybe acquired at discounted prices or freely only in the framework of government--funded projects. Subsequent commercial use and presentation/visualization of these data is not foreseen by the data suppliers. Difficulties in data acquisition arise particularly in regions where the infrastructure of governmental geophysical data is only poorly developed. Data compilation has to rely on support from related external information sources.

#### **Development Status and Practical Experiences:**

Data are supplied in varying quality, attribution and with different geospatial references, which makes their processing necessary.

#### Alternative Procedures:

In the case of incomplete data basis, a literature evaluation on the regions classified as promising regarding mine dumps has to be carried out in parallel, in order to achieve a complete profile of the situation of anthropogenic deposits. Further, a general keyword search in relevant literature databases (e.g. Scifinder, Science Direct, Springer Link, Google Scholar) may be performed. The research is complemented by additional keyword queries in overall search engines (such as Google). Only documents that are clearly identified as publications of scientific origin will be included in the evaluation. These cover articles in scientific magazines, PhD theses and contributions to conferences, publications by associations, as well as legal documents.

Inhomogeneous data formats and detail information due to various data suppliers (merging with metadata) may complicate the data processing. The data quality and the following results are therefore depending on the area and also on the responsible processing institution. For the use of commercial and public geodata, the relevant terms of use apply.

1 Detection and Modelling of Mine Waste Dumps		
1.1 Localization of Dumps	1.1.2 Smoothing Approach	

Authors: Adrian Klink, Sebastian Teuwsen

Objectives:

Detection of dump geometries in unknown areas using digital elevation models (DEM) based on data from ASTER GDEM v2 and differentiation between anthropogenic and geogenic formed ground surface. The principle of the method involves the formation of various smoothing levels using different filter parameters and subtraction of two subsequently applied stages. Local elevations can be detected using the threshold method.

#### Method description:

The smoothing approach implies an attempt to smooth the image surface with two smoothing filters to such an extent that both smoothing processes are distinguished only by the resulting dump contours. The original elevation profile is smoothed with a filter mask. The simplest approach is to use a square averaging mask (of e.g. 3 x 3 or 5 x 5 pixel), which was applied for the described method first. Alternatively, a Gaussian or morphological gray-scale filter can be used as well (dual rank, comparable with Median Filter). Different smoothing levels are subtracted from each other, whereby a stronger smoothed level is always subtracted from the less smoothed one. The difference signifies the local elevations. A simple threshold operator separates the supposedly detected dump from the surrounding area. The estimated threshold value represents the minimum difference in height between both smoothing stages (in meters). However, the threshold value does not represent the height of the dump, and may deviate widely from it, since the smoothing levels vary greatly depending on how strongly the filter mask lowers or elevates the ground level.

Result = Threshold (mean (mask A) – mean (mask B))





Figure: Development of a smoothed ground surface model based on ASTER-Data (screenshot above: processed data, screenshot below: unprocessed data). ASTER Data: © NASA/GSFC/METI/ERSDAC/JAROS, and U.S./Japan ASTER Science Team.

#### Field of Application:

Detection of dumps using satellite-based DEM based on elevation values and separation of stockpiles from not-stockpiles.

**Operational Conditions and Limitations:** 

The parameters for the smoothing masks are area-specific and data-sensitive and can be therefore transferred only with certain restrictions.

**Development Status and Practical Experiences:** 

The approach was prototypically implemented and is operational with minor restrictions. However, it should be noted, that this approach requires permanent adjustments of the parameter sets when applied for new areas.

Alternative Procedures:

Gaussian smoothing instead of the mean value mask; see also the Laplacian-of-Gaussian (LoG) approach.

References / Further Information:

The parameter sets must be checked when the approach is applied for new areas or by transferring data to other satellites (e.g. Sentinel 1 DEM).

1 Detection and Modelling of Mine Waste Dumps		

#### 1.1 Localization of Dumps

1.1.3 Modified Smoothing Approach

#### Authors: Adrian Klink, Sebastian Teuwsen

#### Objectives:

Detection of the dump geometries using digital elevation models (DEM) based on data from ASTER GDEM v2. The minimum from the elevation profile and its smoothed image is calculated several times in succession to determine the soil profile. Subsequently, the relative elevations are invoiced by subtracting the soil profile from the elevation data.

#### Method description:

Similar to the technique of the smoothing approach the image is smoothed via the  $31 \times 31$  mask (ca. 800 m x 800 m). Subsequently (instead of subtraction) the minimum is calculated from the elevation profile and the smoothed image. The areas in the smoothed image which are elevated through the smoothing process are then filtered out with the help of the original image and the soil profile is extracted. The step is repeated twice to obtain the approximation of the soil profile (triple minimum smoothing).

The soil profile is then subtracted from the original elevation profile in order to calculate the relative ground level elevation. The gradient image (first derivative of Gaussian filter) is summed up in the result to sharpen the dump contours. In the next step, the thresholds between 25 m and 20 m are extracted via the hysteresis function (the dumps are slightly super elevated through the gradient image).



Figure: Apply of derivation of Gaussian to ASTER GDEM V2-Data. ASTER-Data: © NASA/GSFC/METI/ERSDAC/JAROS, and U.S./Japan ASTER Science Team.

A check of several different parameter sets – similar to the smoothing approach – is not yet done. The results are based on the parameter set which is subjectively rated as good (as described above). The triple use of the minimum and mean value formation with 31 x 31 mask creates a strongly smoothed soil profile with several erroneous detections in aquatic areas (the water mask should be applied - where possible – to get the correct results). Such errors can be avoided by the prior interpolation and subsequent subtraction of the soil profile from the elevation profile. The result of the first derivative of Gaussian (the gradient image) is used for sharpening the contours of the dumps and other soil elevations. The gradient image depends on the image resolution. A resolution independent approach can be achieved through normalization or alternatively scaling with the factor of the difference between the vertical (height) and horizontal resolution.

Field of Application:

Dump detection using elevation data provided by the satellite-based DEM.

**Operational Conditions and Limitations:** 

The amount of erroneous detections is increased in strongly relief-like areas, since this approach cannot adapt the choice of parameters to a landscape as precisely as the smoothing approach. Nevertheless, the described approach is generally better resistant when applied to new areas.

**Development Status and Practical Experiences:** 

The approach was prototypically implemented and represents a good complement to Harris approach, since it allows to recognize the dumps with no detected significant points and thus to close the gaps on the profile image.

Alternative Procedures: -

References / Further Information:

Canny, J. (1983): Finding edges and lines in images. Report, AI-TR-720, M.I.T. Artificial Intelligence Lab., Cambridge, MA.

1 Detection and Modelling of Mine Waste Dumps		
1.1 Localization of Dumps	1.1.4 Harris Approach	

Authors: Adrian Klink, Sebastian Teuwsen

#### **Objectives:**

Detection of dump geometries using DEM (digital elevation models) based on data from TanDEM-X. The principle of this approach involves finding distinctive points with the Harrisoperator and subsequent shifting of these points to the local maximum. Based on these data, a contour line analysis is carried out with the soil profile as break-off criterion (abrupt surface elevation and compactness measure).

#### Method description:

Preliminary information for positioning of the potential dumps is obtained for higherresolution data (e.g. provided from TanDEM-X) through searching for distinctive points (the Harris-operator) and shifting them to the local maximum. On the basis of this preliminary information an elevation-threshold approach is applied to calculate the dump area to the base point of the dump (similar to Region Growing). The Harris-operator is a combined approach consisting of corner and edge detector, which represents a further development of Moravec's corner detector [s. C. HARRIS, M. STEPHENS 1988]. The distinctive points frequently indicate breaking edges or local elevations and not the highest point of the assumed dump, which is why the points should be monotonically moved to the highest point of the area. Starting from this highest point, an analysis of the elevation levels lying below the highest point is carried out using the threshold approach.

The essential difference between the described approach and the hightlines analysis method is that the shifting of distinctive points to the local maximum already builds a starting point for a potential dump. Thus, the search for the surrounding closed rings of equal elevation is no longer needed to detect a dump. This enables a better graduated contour line analysis, since one can take less consideration of the local interference factors on the contour data / height data. If a potential dump is initialized from several Harris-points and these lead to multiple starting points through their shifting to the local maximum, the duplicates can be avoided while checking whether one such point lies in an already detected dump and thus can be rejected / abolished. The following break-off criteria are chosen for the thresholding approach: a very high value for compactness and a relative surface increase by a factor higher than ten.

#### Field of Application:

Dump detection through the satellite based DEM-contour data.

#### **Operational Conditions and Limitations:**

Ideally, the approach should be applied to the smoothed contour data (noise reduction) or flat soil profile and not to strongly relief-like areas. Among the disadvantages of the approach are the fallacies caused by the dumps situated on slopes. Another disadvantage is that the parameter for generating the Harris-points is data-dependent and might produce too few or too many starting points. In case of too few starting points, the dumps might be overseen or the problem of the false negatives arises. If there are too many starting points, one might come across such problems like too long calculating times for analysis or false positive values for water surfaces, industrial buildings or natural rock formations.

#### **Development Status and Practical Experiences:**

The approach is prototypically implemented and is fully operational with minor restrictions.

#### Alternative Procedures:

As an outlook to further development of the approach the following measures should be mentioned: an analysis of the spectral information or the external classification results (eCognition) to modify the parameters for the analysis/filtering of the dumps. Combined with another alternative approach, e.g. the modified smoothing approach (minimum smoothing for the detection of the ground level), for the sake of reducing the false negative values (e.g. on slopes), the Harris approach represents a promising approach for the automated detection of dumps in unknown areas.

#### References / Further Information:

According to the current state of knowledge the Harris approach is the preferred method for dump detection based on the TanDEM-X data.

References:

Harris, C., Stephens, M. (1988): A combined corner and edge detector. Proceedings of the 4th Alvey Vision Conference, pp. 147-151.

Gouet, V., Boujemaa, N. (2001): Object-based queries using color points of interes. IEEE Workshop on content-based access of image and video libraries, CVPR/CBAIVL 2001, Hawaii, USA.

1 D	1 Detection and Modelling of Mine Waste Dumps		
1.2	Visualization and Modelling	1.2.1	3-D Modelling

Authors: Jochen Nühlen, Asja Mrotzek-Blöß, Michael Jandewerth

#### **Objectives:**

Additional information on the spatial distribution of materials from the production process in the mine dump using selected elevation models. In particual the research and implementation of historical maps / cartographical material for the description of the natural ground surface as basis of the deposit. Visualization of the anthropogenically formed ground surface based on an up-to-date ground model. Intersection of both data in 3D software for visualization.

#### Method description:

The model of the natural (pre-industrial) ground surface has to be georeferenced on the basis of historical maps. Subsequently, after georeferences have been entered, the contour lines (lines of equal elevation) registered in the topographical map have to be digitized and the line data have to be attributed to the Z coordinates (height information). Updated digitized information on ground surface including the relevant X, Y and Z coordinates can be acquired from the responsible agencies by ordering a private laser scanner flight over the dump or by freely accessible data from satellite recording.

The position of the dump on the natural ground surface is produced by combining both 3D models. In successive steps, the generated 3D model can be complemented with further details such as point and ground data. This way, drilling points for example can be entered and visualized. Furthermore, the visualization of previous deposit areas on the basis of detailed maps allows entering the position of filling zones of mono-fractions.



Figure: Left: 3D-Visualization and combination of a current elevation model of the dump site and an elevation model of the 1950-ies, based on historical maps (blue). Right: 3D-Visualization of the current elevation model positioned on the modelled natural ground surface based on historical maps (black). (Screenshot from ESRI ArcScene Software, superelevated 6x, Database of DEM © GeoBasis-DE/BKG

#### Field of Application:

Preliminary analysis of further exploration, site analysis

**Operational Conditions and Limitations:** 

Due to the often-found low resolution of the historical maps and many lacking fix points, no current accuracy standards of a georeferenced topographical map can be assumed.

Exemplarily implemented for a model site.

#### Alternative Procedures:

Evaluation of aerial stereo photographs and conversion of the generated point clouds into a 3D software.

#### References / Further Information:

Some of the cartographic material is based on outdated measuring units (e.g. Prussian foot measure), which have to be converted to the current metric system. In some cases, it helps having the dump visualized in a higher dimension. Depending on the availability of cartographic material from different sections of the deposit history, the combination of the single surfaces allows conclusions on already excavated areas and changes in volume.

#### 1 Detection and Modelling of Mine Waste Dumps

1.2 Visualization and Modelling

1.2.2 SKUA-GOCAD™ 2013.1

#### Author: Lara Satge

#### Objectives:

Computation of multiple data: wells, measured properties (concentrations in elements, particle sizes etc...), layering, topographic surfaces, limits of the tailing etc. in order to get an interpolated/simulated distribution of elements of interest within a 3D Model of the tailing.

#### Abstract:

Importation of all the available data for the tailing is necessary to create the 3D model. Well data are imported as Column-Based File objects for their locations, paths and markers. Each property is associated to the corresponding absolute depth and compiled in a text file. The use of QGIS Software allows to import the properties as an ArcView Shapefile object in Gocad by converting the text file as a shapefile. The properties are then represented in the 3D Viewer as a Point Set where property points appear along the wells' paths.



In order to create a volume where the required properties will be interpolated/simulated, the limits of the tailing are defined by creating the corresponding surfaces. Several steps are compulsory in order to get a proper volume. First of all, the creation of a Voxet respecting the borders of the tailing. This is done thanks to the "From object Box" command. Then, a Stratigraphic Grid (SGrid) is created with a proper resolution regarding the initial data. This 3D grid is fitted between two boundary horizons to model the volume of the tailing and allows to model properties from the Point Set. The properties are created in the SGrid with the "Adding property Control Points" command. This creates all the properties which exist in the Point Set object into the SGrid one. Before any interpolation, the property of interest must be initialized in the grid.

Note: The "Interpolate Property" command is an automated interpolation by Gocad, in order to choose any of the parameters for the interpolation/simulation, a "Reservoir Properties" Workflow must be opened.

#### Field of Application:

3D Modelling for Geosciences: Ore characterization, Reservoir modelling, etc...

Operational Conditions and Limitations:

SKUA-GOCAD<sup>™</sup> 2013.1 is not always well supported and it is safer to keep several versions of the ongoing project. In addition to that, the "Undo" command is not available for lots of actions.

Development Status and Practical Experiences:

SKUA-GOCAD<sup>™</sup> 2013.1 - Paradigm<sup>®</sup> 2011.3 is in permanent evolution and development. It is used by worldwide majors of the Oil&Gas Industry.

Alternative Procedures:

Use of the "Structure and Stratigraphy" Workflow SKUA-GOCAD<sup>™</sup> 2013.1 in order to be guided all along the grid creation.

Use of other Modeling Softwares such as Petrel (Schlumberger), Jewel Suite (JOA), RMS (Roxar).

Characteristics / Remarks:

Advantages of the method:

- wide variety of data from different origins and characteristics can be computed/exploited in one project
- quick and accurate modeling for the required geological application
- control of the geostatistics' tools for the interpolation/simulation of the element of interest

References / Further Information:

Help and Content for SKUA-GOCAD<sup>™</sup> 2013.1 - Paradigm® 2011.3 With Epos® 4.1 Data Management <u>https://pdgm.custhelp.com/</u>

https://tu-freiberg.de/fakult3/gy/mageo/de/skua-gocad

1 Detection and Modelling of Mine Waste Dumps					
1.2	Visualization and Modelling	1.2.3	Reflectance spectral anal	spectroscopy ysis	and

Authors: Michael Denk, Cornelia Gläßer

#### Objectives:

Spectral characterization of dump site materials, derivation of qualitative and quantitative parameters. Reflection spectrometric field and laboratory measurements are non-invasive methods that are used to identify material-specific spectral signatures of different dump site materials. In many cases the spectra allow the characterization of dump site materials as well as the deduction of qualitative (e.g. mineral phases) and quantitative-material parameters.

#### Method description:

Quasi-continuous material-specific spectral signatures are recorded with the help of spectroscopic field and laboratory measurements. The covered wavelength range typically includes the visible light as well as the near and shortwave infrared (350 – 2500 nm). Information on mineralogical characteristics can be by analyzing specific absorption bands. A small number of samples are already sufficient to conduct qualitative and semi-quantitative analyses regarding the connection between the spectral properties (degree of reflectance, absorption band characteristics) and different material parameters, such as the metal content. The mathematical-statistical modelling and estimation of chemical parameters, from spectral data with higher accuracy is carried out by means of chemometric approaches (e.g. PLSR models) using a sufficiently large number of spectra and analyses in order to create a calibration model.

#### Field of Application:

Characterization of materials, qualitative and quantitative material analyses, initial examinations, in-situ analyses

#### **Operational Conditions and Limitations:**

The method is suitable for application in field and laboratory. Under field conditions with varying weather conditions, contact probes with an internal light source may have to be used. Certain by-products of the iron and steel production contain silicates, which do not show specific absorption bands in the wavelength range 350 – 2500 nm and can thus not be spectrally identified.

#### **Development Status and Practical Experiences:**

Within the REStrateGIS project comprehensive systematic reflectance measurements and analyses of by-products from the iron and steel industry taken from the model dump site were carried out for the first time. Reflectance spectra of a number of historical and selected current by-products were acquired and compiled in a spectral library. This library allows typical signatures to be assigned to different dump materials. Furthermore, the spectral identifiability of different mineral phases was examined. The spectroscopic methods for insitu screenings at dump sites were successfully demonstrated at various excavations. Here, materials that appear similar to the human eye could be different in the field turned out to be identical by-products using spectral methods (DENK et al., 2015). Mathematical-statistical relationships between spectral characteristics and chemical parameters could be proven for individual parameters by means of chemometric approaches.

Sampling with subsequent geochemical and mineralogical laboratory analytics are an alternative to the contact-free approach using reflectance measurements. The thus obtained results are potentially more precise, however, the approach is much more cost-intensive and time consuming and not suitable for in-situ screenings.

#### References / Further Information:

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Denk, M., Gläßer, C., Kurz, T. H., Buckley, S. J., Drissen, P. (2015): Mapping of iron and steelwork by-products using close range hyperspectral imaging: A case study in Thuringia, Germany. European Journal of Remote Sensing, Vertical Geology Conference VGC-14 Special issue, 48: 489-509, doi: 10.5721/EuJRS20154828.

1 Detection and Modelling of Mine Waste Dumps			
1.2 Visualization and Modelling	1.2.4	Hyperspectral remote sensing - REStrateGIS Approach	

Authors: Michael Denk, Cornelia Gläßer

#### **Objectives:**

Areal qualitative and quantitative analyses of dump site materials, mapping of the spatial distribution of different dump site materials and their properties. Hyperspectral remote sensing takes up the principle of spectroscopic reflectance measurements at field and laboratory scale (c.f. article »reflectance spectroscopy and spectral analysis«) and transfers it to imaging pixel-based systems. This enables areal qualitative and material quantitative analyses.

#### Method description:

Analogous to field and laboratory spectrometers, hyperspectral imagers record light that is reflected by surfaces in a multitude of narrow bands (up to several hundred). Depending on the sensor properties, the recorded wavelength range covers approximately 400 – 1000 nm or 400 – 2500 nm. In contrast to multispectral data, each pixel in hyperspectral datasets contains an almost continuous spectral signature. The recorded spectra are material-specific and allow the differentiation of various (dump site) materials as well qualitative and quantitative analyses. Hyperspectral scanners are airborne or are – as a comparatively new field of application – terrestrially employed, in particular to answer questions in the fields of mining and geology (e.g. Denk et al., 2015, Kurz et al., 2013, Murphy & Monteiro 2013).

#### Field of Application:

Detection and material analysis of economically relevant materials at embankments and cross-sections by means of imaging terrestrial spectroscopy as well as analyses of surface materials on uncovered dump site (areas) with hyperspectral aircraft scanner data.

#### Operational Conditions and Limitations:

Terrestrial hyperspectral images are particularly suitable for the recording of already existing or new outcrops. Here a good exposure of the sites to the sun is important since the recording systems work passively and record the sunlight that is reflected by the surfaces under field conditions.

#### **Development Status and Practical Experiences:**

Within the REStrateGIS project terrestrial hyperspectral images were taken at selected spots on a dump site from the iron and steel industry for the first time. It was possible to spectrally differentiate various current and historical by-products of different material contents, e.g. varying iron content DENK et al., 2015). Furthermore, the spatial distribution of the by-products found at the surface of the dump site and covering materials were recorded by means of hyperspectral aircraft scanner data.

#### Alternative Procedures:

Comprehensive raster sampling followed by chemical and mineralogical analyses and subsequent interpolation of the selective results by means of geostatistics.

References / Further Information:

Denk, M., Gläßer, C., Kurz, T. H., Buckley, S. J., Drissen, P. (2015): Mapping of iron and steelwork by-products using close range hyperspectral imaging: A case study in Thuringia, Germany. European Journal of Remote Sensing, Vertical Geology Conference VGC-14 Special issue, 48: 489-509, doi: 10.5721/EuJRS20154828.

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1 Detection and Modelling of Mine Waste Dumps		
1.2 Visualization and Modelling 1.2.5 Material flow models		
Authors: Jochen Nühlen, Asja Mrotzek-Blöß, Michael Jandewerth		

#### Objectives:

Creation of a knowledge base on deposited materials and their technical origin on site level and the identification of areas that justify a geotechnical exploration and the subsequent depletion of valuable materials. Elaboration of historical and present material flow models that describe the cause for the genesis of the mine dump as anthropogenic deposit.

#### Method description:

The reconstruction of historical material flows is an essential element of the detailed analysis of an identified anthropogenic deposit. Like for a primary deposit, details on the genesis of anthropogenic deposits allow conclusions on the expected material regarding potential valuable elements and positive or negative side elements. The material flow models are generated from reliable literature on the site and archive data of the company that has produced the deposit by its production activities. In particular, the input and output flows of the production process on the site are visualized over the entire production time period.

If possible this information is complemented and validated by expert knowledge and through inquiries of ex and current staff of the site. The genesis of anthropogenic deposit is thus reconstructed in as much detail as possible, thereby allowing the user to make conclusions about the material to be expected.

Field of Application:

Preliminary analysis, site analysis

**Operational Conditions and Limitations:** 

As experience has shown, the degree of detail and the completeness of the models depend on archive and literature data available on the site or of the company previously or currently active at the site.

#### **Development Status and Practical Experiences:**

Material flow models are worked out for a model mine dump; the method has been successfully tested

#### Alternative Procedures: -

References / Further Information:

The integration of expert knowledge from company staff of the site is of great advantage at facilities that are still operated/active. Ideally, the information from the material flow models is used as basis for detailed analysis and geotechnical investigations on the mine dump. Digging, drilling and geo-physical and -electrical exploration methods help validate the material flow models and further develop them under given circumstances. Combining the production volumes with the material flow models, first conclusions about the volume of anthropogenic deposits expected in the dump can be made.

2	2
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1 Detection and Modelling of Mine Waste Dumps		
1.3 Geophysical Exploration	1.3.1 Spectral Analysis of SIP data	
Authors: Dr. Tina Martin, Dr. Ursula Noell		
<u>Objectives:</u>		
Predict information about location and dimen residual material in abandoned mining dumps	ision as well as the mineral content of waste s.	
Method description:		
The spectral induced polarization method (SIP) is an extension of the conventional geoelectrical method and explores the frequency dependence of the electrical properties. This frequency dependence has proved to be a valuable diagnostic parameter of soils, rocks (e.g. Olhoeft, 1985; Börner et al., 1996; Kemna et al., 2004, Weller et al., 2010, 2013), and ores (Pelton et al., 1978; Kretschmar, 2001). Using a configuration factor that results from the geometry of the arrangement of current and potential electrodes the amplitude of resistivity $ \rho $ is measured in $\Omega$ m. The phase angle $\varphi$ , resulting from the phase lag between current and voltage signal, is usually specified in mrad. The typically used frequency range is between 1mHz and fewkHz. Due to the very time consuming low frequencies and to the limitation of the field devices, the frequency range in field is often between 0.1 and 1000 Hz.		
SIP can be measured in laboratory as 4-p electrode array. For field measurements it is data (apparent resistivity and apparent phase of the structures by mathematical inversion. A is normally used and separate models are co inversion attempt adjacent frequencies are c the inversion. This can be done sequent Moreover, a certain spectral behavior such as implemented into the inversion algorithm. The	point-method and in the field using a multi necessary to reconstruct from the measured e) along a profile the correct place and depth an iterative approach to fit the data to a model postructed for each frequency. In a combined constrained to each other in order to stabilize ially or simultaneously for all frequencies. Is constant phase or Cole-Cole models can be a most general way is to discretize the spectral	

#### Field of Application:

To detect and estimate underground structures from surface SIP measurements it is necessary to analyze not only resistivity and phase for each frequency but also to get information about the chargeability of the subsurface. Particularly the occurrence and dimension of natural mineral deposits can be detected excellently if depth, size, and chargeability contrast of the deposit is within a detectable range. But also for other highly polarisable buried material this method is suitable. An estimation of the mineral's grains sizes from the field data can also be possible since the relationship between the relaxation time and the mineral grain size could be proved (Hupfer et al., 2016).

content of the subsurface model cells using a smooth spectral chargeability distribution

based on the Debye relaxation model (Günther & Martin, 2016).

#### **Operational Conditions and Limitations:**

To analyze spectral IP data the data quality has to be very good. Therefore it is important to use different electrodes for the current injection and the potential measurements (unpolarisable electrodes). Furthermore the frequency range has to be as wide as possible and at least 10 frequencies should be measured.

#### **Development Status and Practical Experiences:**

At the slag heap Kanstein few SIP profiles in a frequency range between 0.1 Hz and 1000 Hz were measured. The example profile below is 39 m long with an electrode separation of 1 m. It was measured using the multi-channel instrument SIP256C applying a dipoledipole array and the above mentioned frequency range. The data were initially inverted independently for each frequency and a significant difference in resistivity and phase could be observed. Afterwards a simultaneous constrained inversion for all frequencies was carried out. Next, the inverted spectra of each model cell were picked and fitted by a Cole-Cole model. As a result a distribution of chargeabilities m and time constants  $\oplus$  is obtainable (Figure).

The result shows a distinct high chargeability (0.7) zone (slag body) in 2-4 m depth. On top and below this zone the chargeabilities decrease. The slag body is also characterized by high time constants (1-3 s).



Figure: Distribution of chargeability (top) and time constant for a SIP profile at a slag heap (Günther & Martin, 2016).

#### References / Further Information:

Günther, T. and T. Martin (2016): Spectral two-dimensional inversion of frequency-domain induced polarization data from historical mining slag heap. Journal of Applied Geophysics, 436-448.

Hupfer, S., Martin, T., Weller, A., Kuhn, K., Günther, T., Djotsa, V. and U. Noell (2016): Polarization effects of unconsolidated sulphide-sand-mixtures. Journal of Applied Geophysics, 456-465.

Kemna, A., Binley, A. and Slater, L. (2004): Crosshole IP imaging for engineering and environmental applications. Geophysics, 69, 97-107.

Kemna, A. et al (2012): An overview of the spectral induced polarization method for nearsurface applications. Near Surface Geophysics, 10, 453-468. Börner, F., Schopper, J. and Weller, A. (1996): Evaluation of transport and storage properties in the soil and groundwater zone from induced polarization measurements. Geophysical Prospecting, 44, 583-601.

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Pelton, W., Ward, S., Hallof, P., Sill, W. and Nelson, P. (1978): Mineral discrimination and removal of inductive coupling with multifrequency IP. Geophysics, 43, 588-609.

Weller, A., Slater, L., Nordsiek, S. and Ntarlagiannis, D. (2010): On the estimation of specific surface per unit pore volume from induced polarization: A robust empirical relation fits multiple datasets. Geophysics 75, No. 4, WA105-WA112.

#### 1 Detection and Modelling of Mine Waste Dumps

#### 1.3 Geophysical Exploration

1.3.2 SIP Laboratory methods

#### Authors: Dr. Tina Martin, Dr. Ursula Noell

<u>Objectives:</u> Predict grains sizes and concentrations of certain minerals in the waste residual material of abandoned mining dumps.

#### Method description:

The spectral induced polarization method (SIP) is an extension of the conventional geoelectrical method and explores the frequency dependence of the electrical properties. This frequency dependence has proved to be a valuable diagnostic parameter of soils, rocks (e.g. Olhoeft, 1985; Börner et al., 1996; Kemna et al., 2004, Weller et al., 2010,

2013), and ores (Pelton et al., 1978; Kretschmar, 2001). Using a configuration factor that results from the geometry of the arrangement of current and potential electrodes the amplitude of resistivity  $|\rho|$  is measured in  $\Omega$ m. The phase angle  $\varphi$ , resulting from the phase lag between current and voltage signal, is usually specified in mrad. The typically used frequency range is between 1 mHz and fewkHz.

SIP can be measured in laboratory as 4-point-method or in field in a multi electrode configuration. Depending on the measurement different analysis methodologies are possible. For the laboratory data the spectra can be fitted to different models (e.g. Cole-Cole or Debye). As results the chargeability m and the relaxation time T can be obtained.

#### Field of Application:

Formerly the induced polarization (IP) method was predominantly used for exploration of mineral deposits due to the strong IP response of the associated minerals. Based on the increasing improvements of the instruments and analysis tools, SIP is nowadays also used in the field of hydrogeological and environmental studies (Kemna et al., 2012) such as detection and demarcation of buried dumps, subsurface lithological definition, hydraulic permeability estimation or hydrocarbon contamination.

#### **Operational Conditions and Limitations:**

To measure SIP in the laboratory a highaccuracy impedance spectrometer (example: Zimmermann et al., 2008) and an adequate 4-point-measuring cell system (example: Figure 1) is necessary. The sample must be inserted into the measuring cell and if applicable saturated by a defined fluid. The current is injected on two points (here: on top and bottom) of the cell whereas the potential can be measured separately (here: on both sides of the inner To avoid temperature effects, cell). measurements have to be prepared under constant temperature conditions (climate chamber). The frequency range in laboratory is often limited between 1 mHz and a few kHz.



Figure 1: BGR 4-point-measuring cell system.

The measurement of the complex resistivity has become a common method in specific areas. For the investigation of historical mining dumps different mineral-sand-mixtures were prepared and measured in order to investigate the method's potential to detect and distinguish different minerals, their grain sizes and concentrations. The results show distinct resistivity differences and phase shifts (Figure 2). As finer the mineral material as higher the phase effects. It could also be seen that with increasing mineral concentration the phase effect increases.



Figure 2: Results for a SIP measurement of a pyrite-sand-mixture with different grain sizes

After fitting the data (spectra) of all measurements to a Cole-Cole model, relationships between the chargeability and the relaxation time are obvious (Figure 3 and see Hupfer et al., 2016). Distinct differences between the three used minerals pyrite, galena and sphalerite could not be observed.



#### References / Further Information:

Hupfer, S., Martin, T., Weller, A., Kuhn, K., Günther, T., Djotsa, V. and U. Noell (2016): Polarization effects of unconsolidated sulphide-sand-mixtures. Journal of Applied Geophysics, 456-465.

Kemna, A., Binley, A. and Slater, L. (2004): Crosshole IP imaging for engineering and environmental applications. Geophysics, 69, 97-107.

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Knödel, K., Krummel, H., Lange, G. (Eds.) (1997): Handbuch zur Erkundung des Untergrundes von Deponien und Altlasten – Geophysik. Voume 3, Springer Verlag, 1075 p.

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Zimmermann, E., Kemna, A., Berwix, J., Glaas, W., Muench, H. M. and Huisman, J. (2008): A high- accuracy impedance spectrometer for measuring sediments with low polarizability. Measurement Science and Technology, 19, 105603 (9 pp).

1 Detection and Modelling of Mine Waste Dumps			
1.3 Geophysical Exploration	133 Combining GPR and ERT data		

Authors: Dr. Rudolf Knieß, Dr. Ursula Noell

#### **Objectives:**

Investigation of the inner structure of the mining dump. This is important for the estimation of the approximate volume of potentially reusable mining deposits within the waste dump.

#### Method description:

Due to the inhomogeneity of the sites we applied electrical resistivity tomography (ERT) and ground penetrating radar (GPR) for detailed investigation. Using ERT we could distinguish various layers within the mining dumps. The resistivities of the dumped material differ from the bedrock resistivities at both sites. The GPR results (layer boundaries) were included into the ERT inversion algorithm to enable more precise and stable resistivity models. This needs some special preprocessing steps. Reflectors detected in the radargram are adopted as resistivity boundaries (interfaces) into the start model of the geoelectric inversion algorithm. This procedure permits the inversion algorithm to find a model with abrupt resistivity changes at theses interfaces (no coupling, no smoothness constraint). To include all boundaries with presumably contrasting resistivities requires numerous different ERT inversion tests.

#### GPR (Ground penetrating Radar)

Is an electromagnetic pulse reflection method based on physical principles. It is a geophysical technique for shallow investigations with high resolution. In its simple time domain form, electromagnetic pulses are transmitted into the ground. A part of this energy is reflected or scattered at layer boundaries or buried objects. The electric field strength E of the pulses (direct and reflected)) are recorded as function of traveltime. Electric permittivity  $\varepsilon$  and electric conductivity  $\sigma$  are the petrophysical parameters which determine the reflectivity of layer boundaries and the penetration depth. The magnetic permeability  $\mu$  is approximately equal to  $\mu_0$  for most rocks. The propagation velocity v, which is important for interpreting depth, is governed to a large degree by the water content of the rock.

#### ERT (Electric Resistivity Tomography)

ERT measurements use DC current or low frequency AC current to induce current into the ground and the generated potential field is measured. The aim is to determine the spatial resistivity distribution in the subsurface. Mainly stainless steel electrodes are used. ERT can be measured along a profile or in a 3D layout on the surface. Depending on the measurement configuration (arrangement of the current and potential electrodes), for example the Wenner array, resolution (sensitivity) and investigation depth vary. To reconstruct from the measured apparent resistivity the "real" resistivity distribution of the subsurface, a mathematical inversion procedure is necessary (Günther et al., 2006). As final results a model of the resistivity distribution along a profile/area is obtained that fits the data best within an error range. This model can give information about the underground structure, e.g. lithology, depth and thickness of aquifers or location and spatial extent of dump materials.

#### Field of Application:

Investigation of abandoned waste dumps and any other kind of sites where heterogeneity is an issue and contrasting resistivities and/or permittivities can be expected. The combination of that two methods helps to locate layer boundaries more precisely. (rock, slags, stamp mill sand, manmade structures, boulders, ...)

#### **Operational Conditions and Limitations:**

The GPR measurements show near surface layer boundaries down to 3 - 4m. The 3D-position of every electrode from ERT measurement and the GPR antenna position on the surface require an accuracy of better than 1 cm. For high precision positioning of electrodes and GPR-antenna a tachymeter and/or dual-frequency GNSS system is required.

At some points, the layer boundaries and radar wave velocities can be calibrated using borehole lithology from a mineralogical drilling campaign. This is important for a precise time-depth conversion of reflectors from GPR measurements. 100 MHz and 200 MHz GPR-Antennas are a good choice for such measurements.

ERT Profiles need electrode distances smaller than 1m for a sufficient resolution. Geoelectrical devices, using more than 50 electrodes, are recommended.

**Development Status and Practical Experiences:** 

Two abandoned small waste dumps in the west of the Harz mountains (Germany) were analyzed. The two investigated dump sites are different in age and therefore differ in their structure. The older residues (< 1930) consist of ore processing waste from density separation (stamp mill sand). The younger dump site comprises slag dump waste. At the first dump site the layer of fine grained residues is less than 6 m thick and the slag layer is less than 2 m thick. Both sites are partially overlain by forest or grassland vegetation and characterized by topographical irregularities.

The Figure shows an ERT inversion result of a profile on top of a thinly layered slag dump. Several different slag types can be separated by their different conductivities.



#### References / Further Information:

Günther, T., Rücker, C. & Spitzer, K. (2006): 3-d modeling and inversion of DC resistivity data incorporating topography Part II: Inversion. Geophys. J. Int.,166, 506-517.

Knödel, K., Lange G., Voigt, H.-J. (Eds.) (2007): Environmental Geology – Handbook of Field Methods and Case Studies. Springer, 1357 p.

REFLEXW, http://www.sandmeier-geo.de/reflexw.html

2 Material Characterization	
2.1 Sample Preparation	2.1.1 Preparation of sample fractions by hydrocyclones

Author: Ulrich Peuser

#### **Objectives:**

Preparation of samples to be treated with different processing possibilities (leaching, flotation etc.)

#### Abstract:

Commonly fractions for test purpose are produced by using test screens. If the interesting cut size is below 20-25  $\mu$ m other possibilities have to be used. Here described is a method using a hydrocyclone.

Depending on the preferred cut size cyclones with diameters of 75 mm and below are used to achieve cut sizes of lower than 20  $\mu m$ :

Cyclone diameter [mm]	Cut size (approx.) [µm]	max. Feed content [g/l]	Pressure loss [MPa]	
75	15	50	0.2	
50	10	50	0.3	
35	7	30	0.3	

For using small hydrocyclones a protection screening is to be recommended at approx. 0.5 to 1.0 mm.

Subsequently the test will be run at a pressure loss and a feed content similar to the table. Simultaneously samples are taken from overflow and underflow fraction over some seconds.



These samples are dewatered in a Büchner funnel. Depending on the further use the samples can be dried completely or stored wet (e.g. for biological purpose). It should always kept in mind that drying changes the behavior of the particle surface.

The grain size can be measured by means of a laser granulometer or a sedigraph or a similar device. Please keep in mind that the different measuring methods cannot be compared!

Field of Application:

Preparation of samples with grain sizes below 25 µm.

**Operational Conditions and Limitations:** 

The cut size of a hydrocyclone is depending on various parameters, mainly particle size, particle density solid content of suspension, cyclone diameter and pressure loss. Of course the separation is not extremely sharp, but imperfections of <0,2 can be reached.

Development Status and Practical Experiences:

Cyclones for low cut sizes are used worldwide in various fields.

Alternative Procedures:

Maybe use of a centrifuge – very similar system

Characteristics / Remarks:

References / Further Information:

Typical literature about processing by means of hydrocyclones as Schubert, H. (2003): Handbuch der Mechanischen Verfahrenstechnik. Several editions available, Wiley-VCH, Weinheim.

2 Material Characterization	
2.1 Sample Preparation	2.1.2 Grain Mounts

#### Author: Dr. Inga Osbahr

#### Objectives:

Mineralogical characterization of tailing material. The material of the tailings is arenaceous with grain sizes up to 300  $\mu$ m. The best way of preparation, for this material with subsequent Mineral Liberation Analysis, are grain mounts.

#### Abstract:

The analyzed sample material has grain sizes from <10  $\mu$ m to 300  $\mu$ m. Material with particle sizes < 20  $\mu$ m requires an additional de-agglomeration step before the usual sample preparation. Thereby, agglomerates in the material are destroyed mechanical and blended with graphite in a volumetric proportion of 1:1. Afterwards, 30 mm round blocks are produced by blending the graphite-sample mix into two grams of low viscose epoxy resin. Air bubbles forming due to steering are reduced by a follow up vacuum system. After the

resin is dried and hardened in a drying furnace, several grinding and polishing steps are applied onto the sample surface to smooth height differences between mineral phases for a level surface. To conclude preparation, a thin layer of carbon is evaporated onto the surface to ensure conductivity of the sample and thus avoid charging of the sample under the electron beam.



#### Field of Application:

Sample preparation for Mineral Liberation Analysis, Mineralogy, Geology, Mineral Processing

Steps	Disk	F in N	time in min	rpm disk	rpm holder	same direction	opposite direction
Grinding							
1	MD-Piano 220	30	2	120	50	х	
2	MD-Piano 1200	25	4	120	50	х	
3	MD-Piano 2000	30	10	100	50	х	
Polishing							
	MD-Dac 550 HC						
4	(3µm Dsp)	25	45	120	50	х	
5	MD-Chem	25	5	40	120	х	
6	MD-Chem	25	5	40	120		х
7	MD-NAP (1µm Dsp)	25	2	120	50	х	
8	MD-NAP (1µm Dsp)	25	2	120	50		X
		(Dsp = diamant suspension)					

#### **Operational Conditions and Limitations:**

Limitations for the sample preparation are the grain size of the material and too large density differences of the phases contained in the material.

**Development Status and Practical Experiences:** 

Sample preparation should provide representative samples. To produce these challenging samples, the method of sample preparation is constantly improved e.g. finding the perfect proportion of material, epoxy resin and graphite. The preparation procedure still has limitations. There are also studies to find the right number of particles for embedding a distinct grain size.

Alternative Procedures:

Strewn samples for samples with large grains sizes and size differences. They are rarely used due to the very complicated and time consuming preparation process.

Characteristics / Remarks:

Advantages of grain mounts

- Very large variety of material can be used
- Large area of analyzed sample surface
- Process relevant grain properties are preserved
- Low costs

References / Further Information:

Lastra, R. and Petruk, W. (2014) "Mineralogical characterization of sieved and un-sieved samples". Journal of Minerals and Materials Characterization and Engineering, 2, pp. 40-48.

Heinig, T., Bachmann, K., Tolosana-Delgado, R., van den Boogaart, G., Gutzmer, J. (2015): Monitoring gravitational and particle shape settling effects on MLA sampling preparation. Extended abstract IAMG 2015, Freiberg.
## 2 Material Characterization

## 2.2 Particle Size Measurement

Author: Dr. Thomas Leißner

#### **Objectives:**

Determination of the particle size distribution and size parameters of drill core segments and of products from different processing steps.

## Abstract:

The particle size distribution of drill core segments is measured to investigate layering of tailings inside the heap body. Layering results from spill points changing over the years. Coarse particles settle fast whereas fine do slow. That is why the coarse particles are enriched near to the spill point and fines are enriched in the center of the tailings pond. Due to the composition of tailings there should be coarse particles which are barren or just contain small amounts of valuables. As separation always is incomplete or particles are too small for upgrading with the state of the art separation technology, the fine fraction contains of liberated valuables and gangue. Thus a layering in particle size and grade results.

Particle size distributions of mill products are measured to study grinding results and to evaluate the feed for downstream classification or separation processes.

If the sample is broad in size distribution, one method of particle size measurement cannot cover the whole range. Therefor different methods have to be used and the results have to be combined. This is a difficult task due to the different size parameters measured by different methods. In analytical sieving particles are compared with the openings of the screen. As long as two dimensions of the particles are smaller than the opening, this particle theoretically can pass the screen. The size of the last opening the particles pass through is used as particle size definition.

Some methods are using equivalent diameters for particle size definition. This equivalent diameter is the size of a sphere (or circle in tow-dimensional measurement) having the same feature like the measured particle. For example the settling speed in sedimentation analysis or the diffraction pattern in laser diffraction.

Note: The shape of particles as well as the density of different phases cannot be considered using methods based on equivalent diameters.

## Field of Application:

Characterization of samples / materials in geoscience, natural science and engineering

## **Operational Conditions and Limitations:**

The operational conditions and limitations are related to the analytical instruments as well as to the composition of the sample and the sampling method. To ensure accurate measurement and to prevent the laser diffraction device for damage, an analytical sieving at 315  $\mu$ m was used before laser diffraction analysis.

## Sampling method

- Samples taking from drill cores using a spoon. The drill core was halved lengthways before sampling.
- Samples taking from buckets using mechanical samplers (cylindrical hollow drill). 5 subsamples were taken at different points of the bucket trying to cover the whole height. The subsamples were combined to one sample for analysis.

Analytical sieving

- Wet sieving before laser diffraction measurement down to 315 μm.
- Standard analytical sieves of 200 mm diameter

Laser diffraction (Sympatec HELOS)

- Wet measurement (SUCELL, CUVETTE)
- Measurement range 0.4 to 800 µm
- Deagglomeration using an ultrasonic sonotrode

**Development Status and Practical Experiences:** 

State of the art method.

Alternative Procedures:

Alternative procedures for particles size measurement are:

- Dry analytical sieving down to 50 µm
- Wet analytical sieving down to 20 µm
- Dry analytical sieving using air jet sieving machines down to a particle size of 5 µm
- Image analysis techniques static or dynamic (flow cell)
- Sedimentation analysis

## Characteristics / Remarks:

It has to be taken into account, that particle size measurements using equivalent diameters contain a bias in mass distribution when samples composed of different phases (densities) are measured.

Furthermore, particle size distributions measured with different techniques e.g. laser diffraction, sedimentation analysis and analytical sieving cannot directly be compared. Nevertheless, they have to be combined in practical use due to the limited range in particle size of the techniques.

References / Further Information:

Rhodes, M. (2008): Introduction to particle technology. 2nd ed., John Wiley & Sons, Ltd, 449 p.

Schubert, H., (2003): Handbuch der Mechanischen Verfahrenstechnik. Several editions available, Wiley-VCH, Weinheim.

2 Material Characterization		
2.3 Geochemical Characterization 2.3.1 Chemical mapping of drill cores with LIBS core scanner		
Authors: Kerstin Kuhn, Jeannet Meima, Dieter RammImair <u>Status:</u> as of 202		
<u>Objectives:</u>		
Chemical mapping of drill cores from mine we enrichment and depletion as well as different the assessment of the metal content in the m	waste sites in order to de t lithological zones. The c ine waste dump.	etect zones of metal lata can be used for
Method Description:		
In Laser Induced Breakdown Spectroscopy optically focused onto a small spot on the vaporizes and excites a small amount of sam causing the atoms and molecules to emit I obtained emission spectrum is, therefore, ch the sample. Element mapping of drill core se which is applicable for point measurements, either the laser is moved over the sample or th biaxial translation stage.	(LIBS), a very short-dui sample surface, genera ple material. The plasma ight at their characteristi haracteristic for the eleme ections can be done by a 1D profiles and 2D scar ne sample is moved via a	ation laser pulse is ating a plasma that expands and cools, c wavelengths. The ental composition of LIBS core scanner, ns. During mapping, computer-controlled
Image: second stage with axis motors         Image: second stage with ax	spectrometer + CCD fibre anner used in a prototype original LIBS 5 and 964 nm. f one drill core bhlfahrt (Harz tBS core	Ag Sb Zn SiO <sub>2</sub> Ba
spectrum contains 36.400 support points between 285 Right: Photo, core log and element distribution maps o meter from the mine waste heap Bergwerkswo Mountains, Germany). Element mapping was done with scanner in combination with a PLS regression model. The layers have grain sizes with predominant gravel/s sand/silt (light green) or silt/clav (green).	5 and 964 nm. f one drill core ohlfahrt (Harz the LIBS core he mine waste sand (yellow), lighest metal	

On the basis of calibration samples with similar matrices, the quantification of emission intensities is possible. Additionally, calibration-free approaches for quantification exist for a number of applications. Quantification is done using pre-processing methods in combination with multivariate statistics or neural networks, or hybrid approaches of different methods. In addition to element mappings, mineral or phase distribution images can also be generated.

concentrations with about 7% Pb, 420 ppm Sb and 180 ppm Ag occur

in the fine-grained material.

## Field of Application:

The LIBS core scanner can be used for fast mapping of large amounts of rock samples or drill cores without any further preparation. It can therefore be applied, for instance, for exploration of ore deposits or mine waste sites, process control in manufacturing or beneficiation of a wide variety of materials, or in research.

## **Operational Conditions and Limitations:**

LIBS scanners can be used in the laboratory as well as in the field or in a production plant. As the temperature of the spectrometer must be kept constant, this can be done either via the room temperature or by extra cooling of the spectrometer. Most disadvantageous are matrix effects, which influence the LIBS intensities. Thus, emission intensities of one element measured in two different sample matrixes do not necessarily represent real element concentrations. The initial quantification of samples with a new composition is, therefore, associated with relatively great effort and can only be carried out by well-trained operators. Once quantification has been established, however, large sample quantities can be analyzed within a very short time and without great effort. For mineral or phase mapping, however, the matrix effects can be advantageous.

## **Development Status and Practical Experiences:**

While LIBS used to be rather limited to material science with homogeneous samples, it is nowadays extensively used also for heterogeneous samples in all fields of application. Applications are in materials science, geoscience and other fields, where they are used in research, exploration of raw materials, process control in recycling, and in industrial manufacturing (Fabre, 2020; Galbács, 2015, Harmon et al., 2021). In addition to commercial LIBS devices, users can configure their LIBS device on their own according to the samples that are investigated.

## Alternative Procedures:

Current core logging techniques include porosity logging (bulk density), multi-sensor core logging (gamma ray attenuation, P-wave velocity, magnetic susceptibility, electrical resistivity, spectrophotometry, natural gamma), digital imaging and non-imaging optical systems, hyperspectral imaging, X-ray fluorescence (XRF) spectral scanning, X-ray computed tomography (3D X-radiography of sediments), laser-induced fluorescence (LIF) spectroscopy, magnetic resonance imaging, and confocal macro/microscopy with laser imagery, as well as combinations of the above technologies. Some of these methods can perform element mapping while others can distinguish specific phases or minerals or map other material properties. Whereas natural gamma-ray spectrometry, for example, allows estimation of K, U, and Th concentrations, XRF is able to detect a wide range of elements from Al to U.

## References / Further Information:

Fabre, C. (2020): Advances in Laser-Induced Breakdown Spectroscopy analysis for geology: a critical review. Spectrochimica Acta Part B: Atomic Spectroscopy, 166, 105799.

Galbács, G. (2015): A critical review of recent progress in analytical laser-induced breakdown spectroscopy. Analytical and Bioanalytical Chemistry, 407, 7537-7562.

Harmon, R.S., Senesi, G.S. (2021). Laser-Induced Breakdown Spectroscopy – A geochemical tool for the 21st century. Applied Geochemistry, 128, 104929.

Kuhn, K., Meima, J.A., RammImair, 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.

2 Material Characterization	
2.3 Geochemical Characterization	2.3.2 Solid Sample Digestion

Authors: Christine Pilz, Stephanie Uhlig

## **Objectives:**

Transfer of solid samples with components of low solubility, e.g., ore and rock, into solutions for quantitative analysis with suitable methods (e.g. Inductively Coupled Plasma - Mass Spectrometry, ICP-MS; Inductively Coupled Plasma - Atomic Emission Spectroscopy, ICP-AES)

## Abstract:

The most common method is acid digestion under pressure: A representative aliquot of homogenized sample material reacts with a mixture of highly purified, concentrated acids. The acids, their volume and concentration are defined by the chemical composition of the sample material.

A digestion system consists of chemically inert vessels, and a heating and vaporization unit. Such systems ensure safe and controllable operation under defined temperature and pressure conditions.

An acid digestion consists of three subsequent steps. First, refractory components are transferred into soluble compounds. An example is the reaction of silicon dioxide with hydrofluoric acid:

## $SiO_2 + 4 \text{ HF} \rightarrow SiF_4 + 2 \text{ H}_2\text{O}$

The obtained silicon tetrafluoride  $(SiF_4)$  is removed from the solution due to its high vapor pressure in the second step – heating and vaporization. In the third and last step, remaining digestion products are dissolved in appropriate, diluted acids. In most cases HNO<sub>3</sub> is preferred because it avoids interferences during ICP-MS measurements.

Following fuming off the acids under controlled conditions and dilution of the sample in ultrapure water, the solution can be now analyzed by several spectrometric methods depending on the elemental composition of the sample. ICP-MS and ICP-AES are available in most geochemical laboratories, providing simultaneous detection of most elements. Often, ICP-OES measurements are sufficient because its detection limits correspond to the profitable concentrations.

Field of Application:

- Quantitative analyses particularly of light elements (Li to F; cannot be detected quantitatively by most XRF), of rare earth elements, and other trace elements (better determination limits than XRF)
- Small sample amounts (upper mg range)
- Complete validation of mining sites by total digestion

## **Operational Conditions and Limitations:**

The analyzed aliquot has to represent the total sample amount that needs to be large enough to realistically represent the site of interest.

For total digestion, the knowledge of the mineralogical composition of the sample is essential. This delivers important information for the acids and the digestion conditions to be employed.

The procedure must be modified if compounds with high vapor pressure (for example SiF<sub>4</sub>) need to be determined quantitatively.

Development Status and Practical Experiences:

Digestion methods have a long tradition. They are established in high-throughput routine analyses as well as in special applications for selected samples. Numerous digestion systems are commercially available. Detailed information can be found in a variety of literature – see "References / Further information" for details.

Several analytical methods are available for elemental analysis. ICP-MS and ICP-AES are just the most common ones.

Alternative Procedures:

- X-Ray Fluorescence (WD-XRF, ED-XRF)
- Ion chromatography (IC)
- Atomic Absorption Spectroscopy, AAS (furnace, F, or electro thermal, ET)
- Fused beads of solid sample material can be dissolved in appropriate solvents and analyzed by known methods.

#### Characteristics / Remarks:

Commercial laboratories often offer packages of total digestion and geochemical analysis, which can be used for a reliable validation of the mining site. In contrast, Aqua regia extracts only provide simple information for exploration. Yet, standard commercial procedures do mostly not consider specific challenges that emerge, e.g. with sulphur-rich ores.

References / Further Information:

Heinrich, H. (1989): Aufschlußverfahren in der Analytischen Geochemie (Teil 1). LABORPRAXIS (12), 1-6.

Heinrichs, H. (1990): Aufschlußverfahren in der Analytischen Geochemie (2). LABORPRAXIS (1/2), 7-10.

Heinrichs H, Herrmann A.G. (1990): Praktikum der Analytischen Geochemie. Berlin: Springer-Verlag, 669 p, doi: 10.1007/978-3-642-61286-2.

Smith, P.L. (2001): A primer for sampling solids, liquids, and gases: Based on the seven sampling errors of Pierre Gy. Philadelphia, PA, Alexandria, VA: Society for Industrial and Applied Mathematics; American Statistical Association.

2.3 Geochemical Characterization 2.3 Geochemical Characterization 2.3 Geochemical Characterization 2.3.3 Acid Extraction for the Determination of the Lithium Content in Tailing Materials Containing Lithium-Bearing Micas	2 Material Characterization		
	2.3 Geochemical Characterization	2.3.3	Acid Extraction for the Determination of the Lithium Content in Tailing Materials Containing Lithium-Bearing Micas

Author: Dr. Michael Scheel

## Objectives:

Chemical characterization of tailing material. Quantitative determination of lithium in form of lithium micas (e.g. zinnwaldite) and other acid-soluble lithium compounds in tailing materials after an acid extraction.

## Abstract:

The analytically fine powdered and dried sample is mixed with a mixture of five units of volume of conc. nitric acid and one unit of volume of conc. hydrofluoric acid in a PTFE beaker. The sample-acid-mixture is heated on a heating plate for one hour after capping with a lid. Subsequently the mixture is inspissated until it is dry. The vaporized sample is taken up with demineralized water and conc. nitric acid and is heated again on a heating plate. The solution is transferred into a graduated flask and is filled up to the nominal volume. Subsequently the solution is filtrated and the lithium content is determined with ICP-OES.

## Field of Application:

Mineralogy, metallurgy, chemistry, raw materials production

**Operational Conditions and Limitations:** 

This method is not a complete pulping of the sample material but an extraction with a mixture of acids. With this method, the acid-soluble lithium contents in particular of lithium micas such as zinnwaldite as well as other acid-soluble lithium compounds are digested.

**Development Status and Practical Experiences:** 

The extraction with a mixture of nitric acid and hydrofluoric acid was operated several times successful at the Institute for Nonferrous Metallurgy and Purest Materials, TU Bergakademie Freiberg for the determination of lithium contents in zinnwaldite concentrates and in tailing materials containing zinnwaldite.

Alternative Procedures: -

Characteristics / Remarks:

- The element rubidium which is associated with the micas can also be determined.
- The extraction is simple to perform.

References / Further Information:

TU Bergakademie Freiberg, Institute for Nonferrous Metallurgy and Purest Materials, http://tu-freiberg.de/fakult5/inemet



# Operational Conditions and Limitations:

calibration and suitable correction methods (software packages).

Matrix-matched calibration of the spectrometer is essential. Detected intensities need to be corrected by different correction factors, which consider the matrix and related interfering effects, for example mass attenuation coefficients. A number of commercial software packages for specific matrices is available, for example for oxides, slags, and trace elements. Yet, nothing is directly suitable for tailing materials since these are highly diverse. Alternative techniques (calibration plus correction software) can operate with a variety of matrices, but the obtained results yield bigger errors. Therefore, commercial labs often

declare, e.g. maximum sulfur contents for WD-XRF-samples to avoid misinterpretations as a result of inadequate calibration.

Detection limits vary in different matrices. For example, there are programs (requiring program-oriented calibration) that can detect trace concentrations (some mg kg<sup>-1</sup>) in rock matrices, but no major components. Other programs (again requiring program-oriented calibration) can determine major components up to 100 wt.-%, for example in oxidic matrices, but no traces. It is not wise to use a program for trace elements to determine major components will be applied in an unsuitable way. Especially trace element programs work only in narrow determination confines. Tailing materials are often composed of "trace elements" that occur in much higher concentrations as in rock material. This higher background of heavier elements such as Ni, Cu, Zn, Sn, W, Pb ..., as compared to Si, influences these mass attenuation coefficients in a way that will lead to wrongly calculated results. If samples show concentrations in the calibration range, then elements from at least fluorine to uranium can be analyzed without problems.

Depending on the purpose, a minimum sample (< 63  $\mu$ m) amount between 1 g and 10 g is required. This relatively high sample mass is needed since the specimen has to withstand vacuum conditions and the powerful X-ray exposure during analysis to guarantee sufficient thickness for the penetration depth of X-rays and to obtain more representative results. There are also techniques to analyze loose powder, but this yields comparatively bad count rates and higher statistical errors. For satisfactory analytical results, it is paramount that the sample material is < 63  $\mu$ m in grain size, homogeneous in itself and well mixed with the binder to obtain a plain specimen surface. Sample fractionation during pressing (pressed powder pellets) may pose problems.

#### **Development Status and Practical Experiences:**

WD-XRF is a common and well-known method for geochemical sample characterization. It is widely used in academia and industry for analysis and process control. Although this method is widely operated, it is very important to be aware of its susceptibilities and constraints.

#### Alternative Procedures:

WD-XRF offers the opportunity to analyze fused glass beads, pressed powder pellets and loose powder. Alternative procedures for chemical characterization of solid samples without previous sample digestion could be TXRF (Total reflection X-ray spectrometry), SS-AAS (Solid Sampling Atomic Absorption Spectrometry) and INAA (Instrumental Neutron Activation Analysis).

#### Characteristics / Remarks:

- High precision of the method, but highly prone to systematic errors (-> matrix effects and correction factors)
- Powdered sample material can be analyzed without previous digestion
- Sample preparation has extremely high influence on quality of analysis
- Higher sample mass leads to more representative results
- Only one calibration needed at setup no further calibration required
- Comparatively fast sample analysis due to a quasi-simultaneous measurement
- Overnight analysis without supervision
- Once a specimen is prepared, it could be measured various times.

#### References / Further Information:

Jenkins, R (1999): X-ray fluorescence spectrometry. John Wiley and Sons Inc. New York; 207 p.

## 2 Material Characterization

2.4 Mineralogical Characterization

2.4.1 X-ray diffraction (XRD)

## Author: Rene Luhmer

## Objectives:

Sample preparation for X-ray diffraction analysis

## Abstract:

For quantitative mineralogical investigations a homogenized, representative, air-dried aliquot of about 2-3 mL of the original material is needed. In case of the existence of easy oxidizing contents the freeze-drying method is recommended for sample drying. To obtain a representative subsample a sample divider is used. The aliquot is then micronized with 10 mL ethanol (depending on the sample with anhydrous ethanol) with the help of a McCrone micronizing mill for 10 min to a mean grain size of 3-7  $\mu$ m. For an optimal milling process the sample should exist as particles smaller than 400  $\mu$ m. In case of the presence of an amorphous content in the samples, the usage of an adequate internal standard, like corundum or zincite, is necessary. This internal standard has to be added with a defined amount to the sample before micronizing. After micronizing the sample is air-dried and subsequently homogenized using a vibratory mixer mill or something similar. Afterwards, the sample is ready to be measured with an X-ray diffractometer.

If knowledge about the dependence of the mineral abundances to the grain sizes is demanded, the samples have to be sieved into appropriate sizes before micronizing. To minimize the formation of dust, it is recommended to sieve the samples with water. If there are minerals like sulfides in the sample, which easily oxidize in contact with water, ethanol is a more suitable choice of sieving medium.

**Operational Conditions and Limitations:** 

The sieving with ethanol is to be performed beneath an exhaust hood.

Development Status and Practical Experiences:

The sieving and the following drying are time consuming procedures.

Characteristics / Remarks:

Despite of the usage of substances (e.g. ethanol instead of water as sieving medium or the usage of a freeze-dryer) to prevent the samples for oxidizing, a small degree of alteration can be possible.

2 Material Characterization		
2.4 Mineralogical Characterization	2.4.2	Mineral Liberation Analyzer (MLA)
<u>Author:</u> Dr. Inga Osbahr		

#### **Objectives:**

Mineralogical characterization of tailing material concerning modal mineralogy and grade of liberation, phase associations, grain sizes of the minerals of interest as cassiterite, wolframite, bismuth, molybdenite, arsenopyrite, sphalerite, galena, etc.

## Abstract:

Grain mounts (epoxy blocks) were prepared for quantitative mineralogical analysis using the MLA software for SEM. For this purpose, aliquots of 3 g of solid material were mixed

with graphite and epoxy resin. Resulting sample blocks (30 mm in diameter) were ground and polished. All samples were carbon coated by use of a Leica EM MED 020.

SEM-EDX measurements were carried out using a FEI Quanta 650F scanning electron microscope equipped with two Bruker Quantax X-Flash 5030 EDX detectors and FEI's MLA Suite 3.1.4 automated for data acquisition. It combines the information gathered



by backscatter electrons (BSE) images and the collection of energy dispersive X-ray spectra (EDX) of particles with a specified contrast in the BSE image.

The collected spectra are classified with a standard mineral spectra list which has been collected and specified for the samples. Resulting data are about chemical composition, qualitative and quantitative mineralogy (with distinct constraints), information on grain sizes and their distribution, phase associations, and the degree of liberation of minerals of economic interest.

#### Field of Application:

Geosciences, Geometallurgy, Mineral Processing, Material Science, Environmental Science, Chemistry

#### **Operational Conditions and Limitations:**

Limitations are the vague differentiation of mineral phases with same chemical composition but various crystal structures (polymorphs) as e.g. pyrite and marcasite or the preparation of materials with several phases which differ in their hardness. This method is also limited by grain sizes < 1  $\mu$ m. No detection of H, He, Li, Be.

SEM parameters		MLA parameters	
Voltage [kv]	25	Measurement mode	GXMAP
Working distance [mm]	12	Scan speed	16
Probe current [nA]	10	Resolution [px]	1k x 1k
Spot size	5.85	Pixel size [µm/px]	0.5
Horizontal Field Width	500	Acquisition time [ms]	6
Brightness	82.8	Quartz EDX-count	2000
Contrast	31.6	Step size [px]	6x6
BSE calibration	Cu 252	GXMAP trigger	25-255
		Min. particle size [px]	4
		Min. grain size [px]	2
		Particle count	>50,000-200,000

## **Development Status and Practical Experiences:**

The current MLA System is produced by FEI and consists of a Quanta 650F scanning electron microscope equipped with two Bruker Quantax X-Flash 5030 EDX detectors and FEI's MLA Suite 3.1.4.

The MLA is used by industry (e.g. Anglo American Platinum, Boliden, Thyssen, SGA) and research institutes (e.g. SGS, Mintec, BGR, TU-Bergakademie Freiberg, Helmholtz Institute Freiberg).

## Alternative Procedures:

Alternative procedures commonly used for mineralogical analyses are e.g. powder X-ray diffraction (XRD), however the detection limits resulting from the MLA are much lower and additional information as listed above are provided.

## Characteristics / Remarks:

Advantages of the method:

- Efficient tool for a large field of science e.g. geoscience, exploration, ...
- Wide range of material can be analysied
- Quantitative and qualitative analysis
- Fast and large area detection
- Non destructive
- Low detection limits for usual mineral phases and materials

## References / Further Information:

Fandrich, R., Gu, Y., Burrows, D., Moeller, K. (2007): Modern SEM-based mineral liberation analysis. International Journal of Mineral Processing, 84, pp. 310-320.

Gu, Y. (2003): Automated Scanning Electron Microscope based Mineral Liberation Analysis. Journal of Minerals & Materials Characterization & Engineering, 2, pp. 33-41.

#### 3.1 Reclaiming Methods

#### Author: Mirko Martin

#### Objectives:

Reclaiming methods aim at excavating the dump material and preparing it for further processing.

#### Abstract:

The technology to be chosen for reclaiming of tailings from the dumps depends strongly on the geotechnical properties of the tailings material and on the geotechnical characteristics of the tailings dam.

A comparison of advantages and disadvantages is given in tabular form. A variant comparison showed advantages of hydromechanical reclamation by Hydromonitor. It can be operated from the accessible rim of the tailings dam.

#### Field of Application:

Reclaiming methods are applicable for extraction of material from tailings and other waste dumps.

## **Operational Conditions and Limitations:**

Operational conditions of the reclaiming technologies depend on the geotechnical properties of the tailings material and on the geotechnical characteristics of the tailings dam. A comparison of advantages and disadvantages of different reclaiming technologies is given in the table.

Technique	Advantage	Disadvantage	
Bucket Wheel Excavator	continuous operation, good cutting effect	low time utilization, frequent retraction	
Universal Excavator	technically mature, high operational safety	accessibility necessary	
Front-End-Loader	good maneuverability, universally applicable	discontinuous operation	
Scraper	technically and operational simple, sectoral operation	decreasing loading performance by increasing scrape distance, difficult handling, frequent retraction	
Dozer	technically mature, high operational safety under rough operational conditions	small range of action (50 m), complex transfer points	
Bottom-Dump Scraper	universally applicable by combination of load and haulage operation	discontinuous operation, no selective reclamation	
Hydromechanical Reclamation (Monitor)	largely continuous operation, pulp parameters directly adjustable to requirements of further processing	high water requirement	
Floating Dredger	continuous operation	open water necessary	

- water saturated very fine grained material
- unsaturated coarser grained material.

According to the deposition technology of the tailings in the dams by pumping, the fine grained material is deposited in the dam center and the coarser grained material on the rim. Frequently the center of the tailings dam is not accessible. Therefore, the reclaiming operations have to be done from the rim of the dam.

Variant comparison showed advantages of hydromechanical reclamation by Hydromonitor. It can be operated from the accessible rim of the tailings dam. The rim has to be reclaimed as reclamation of the fine grained material on the center of the dam proceeds.

Limitations result from legislation (water protection, nature conservation).

**Development Status and Practical Experiences:** 

Historically reclamation of tailings was operated in Altenberg (tailings dam "Tiefenbach") in industrial scale by dredging and hydraulic transport. In SMSB only desktop based pre-investigation of the reclaiming technologies were done.

Alternative Procedures:

Possible alternative procedures involve in situ-leaching operations. Applicability depends on water permeability of tailings material.

Characteristics / Remarks:

References / Further Information:



ball size	$\boldsymbol{d}_{\mathrm{K}} = \mathbf{C}\sqrt{\boldsymbol{d}_{80}} \cdot \left(\frac{\boldsymbol{\rho}_{\mathrm{G}} \cdot \boldsymbol{W}_{i,m}}{\boldsymbol{\psi}}\right)^{1/3} \cdot \boldsymbol{D}^{-1/6}$	in mm
	d <sub>80</sub> 80-%-particle size (feed)	in µm in ka/m³
	$W_{i,m}$ Arbeitsindex nach BOND	in kWh/t
	C 0,024 für Stahlkugeln $\Psi$ relative speed	
	l l	
Mass of ball filling:	$m_{\rm KF} = \frac{\pi}{4} \cdot \left(1 - \varepsilon_{\rm Sch}\right) \cdot \rho_{\rm St} \cdot \varphi_{\rm KF} \cdot D^2 \cdot L \approx 3700 \cdot \varphi_{\rm KF} \cdot D^2 \cdot L$	in kg
	$\varphi_{\text{KF}}$ filling (balls, 0.4 0.45)	
	$\varepsilon_{\rm Sch}$ porosity of ball filling (0.4)	
	D. L mill dimensions	in m
	,	

## Field of Application:

mineral processing, industrial minerals, process engineering

## **Operational Conditions and Limitations:**

## Sample

- Particle size distribution and characteristic parameters (e.g. *x*<sub>90</sub>)
- Liberation distribution
- Content of waste material like organics and metal parts (should be split off the material before grinding)

## Grinding

- Mill dimensions (diameter, length)
- Mill filling (balls, ball size distribution, amount of feed...)
- Rotation speed (% critical speed)
- Solid content (pulp density) in wet grinding
- pH-value (pulp viscosity, pulp conditioning before flotation)
- Grinding time

## Sampling/Analysis

- Samples can either be taken using wet or dry working sample splitters or by putting the pulp into a stirred tank and taking the sample using a syringe.
- The particle size distribution of the product has to be analyzed to evaluate the grinding process in terms of breakage ratio and product size distribution.
- A mineral liberation analysis of the product should be done to evaluate the liberation of minerals, their size distribution and different breakage behavior.

## **Development Status and Practical Experiences:**

State of the art method.

## Alternative Procedures:

Grinding with other types of mills (e.g. agitated ball mill, vertical roller mill).

## Characteristics / Remarks:

The parameters of the mill used in this investigation are listed below.

- mill dimension: 205 mm length, 200 mm diameter -
- rotation: 70 min<sup>-1</sup> (79 % critical speed)
- filling:  $\varphi'_{MG} = 1.0$ \_
- ball size distribution: 40 mm (kg), 25 mm (kg), 20 mm (kg), 15 mm (kg)
- 20 / 30 / 40 wt.% solids pulp density: 7
- pH-value: \_

## References / Further Information:

Schubert, H. (1989): Aufbereitung fester mineralischer Rohstoffe, Volume 1, 4th ed., Deutscher Verlag für Grundstoffindustrie, Leipzig.

Weiss, N.L. (1985): SME Mineral Processing Handbook. Society of Mining Engineers of the American Institute of Mining, Metallurgical, and Petroleum Engineers.

Wills, B.A. (2006): Mineral Processing Technology. Butterworth-Heinemann.

## 3.2 Mineral Processing

3.2.2 Physical separation – Attrition

Authors: Thomas Felbinger, Dr. Sebastian Prinz

## **Objectives:**

Many minerals carry coatings or deleterious materials on their surfaces that need to be removed prior to applying downstream processes. Scrubbing is a process through which mineral surfaces are liberated and deleterious minerals separated. Furthermore agglomerates consisting of several particles are disintegrated.

## Abstract:

Attrition scrubbers are mainly used for washing of material below 10 mm in size. Very high energy inputs are possible.

Attrition scrubbers are simple, yet highly efficient units for scrubbing particles at slurry densities of 70-80% solids. Two opposed Helix impellers on each shaft create an intensive mixing action forcing individual particles against each other resulting in scrubbing, surface cleaning and disintegration of agglomerates.

Scrubbing was used to clean the surface of the particles. Thereby fine particles attached to the surface of the sample are abraded and dispersed in the added liquid. For this treatment, the sample was filled in an attrition cell and stirred intensely with a multi-stage stirrer at a solid content of > 65 wt.-%.

## Field of Application:

Removal of Fe oxide stains from sand particles. Disintegration of clay agglomerates in sand. Delamination of minerals such as kaolin and graphite. Blunging or slurryfying of dry clay prior to wet processing. Oil/Sand separation. Lime Slaking.

**Operational Conditions and Limitations:** 

Operational conditions and limitations for attrition scrubbing are listed below.

## Operational conditions

- Retention time is based on test results. In the absence of any other information assume a retention time of 6-8 minutes at 75% solids w/w for a typical sand scrubbing duty.

## Limitations

- Maximum size of individual particle to scrubber is 10 mm
- Due to the flow pattern an even number of cells must be selected (e.g. 2, 4, 6 cells)

## Development Status and Practical Experiences:

State of the art method.

Alternative Procedures:

Alternative procedures for attrition scrubbing are:

- Log washer
- Vibration screw

Characteristics / Remarks:

Attrition scrubbing is always influenced by the solids content, the particle size and particle size distribution of the feed material. A classification into narrow size fractions before separation will lead to an improvement in the product quality.

## References / Further Information:

Drzymala, J. (2007): Mineral Processing, Foundations of theory and practice of minerallurgy. Wroclaw: Oficyna Wydawnicza PWr.

Schubert, H. (1977): Aufbereitung fester mineralischer Rohstoffe – Sortierprozesse. Volume 2, 2nd ed., Deutscher Verlag für Grundstoffindustrie, Leipzig.

Metso Corporation (2015): Basics in Minerals Processing. 8th ed.

Thaler, P., Walter, B. (2012): Ex-situ Behandlung von kontaminierten Böden – Anlagen in Österreich und angewandte Praxis. Report REP-0390, Umweltbundesamt, Wien, 87 p., ISBN: 978-3-99004-193-2.

## 3.2 Mineral Processing

3.2.3 Selective Fragmentation

Authors: Thomas Felbinger, Dr. Sebastian Prinz

## **Objectives:**

Selective Fragmentation is an electric pulsed power process. High-voltage (HV) discharges are applied for the fragmentation, disintegration or disaggregation of solids.

## Abstract:

Selective Fragmentation is an innovative technology which fragments rocks at a much higher purity than conventional comminution processes, selectively along the grain boundaries of the minerals. Material in a dielectric liquid (water) is exposed to high voltage pulses (ns/kV) (**Fehler! Verweisquelle konnte nicht gefunden werden**.). The electrical discharge travels through the material and generates strongest internal shockwaves. Consequently, composite materials are fragmented along grain boundaries with a high degree of selectivity. Liberated minerals can be selectively separated by post treatment processes e.g. flotation or magnetic separation.



Figure: Principle of selective fragmentation equipment

Selective fragmentation has tremendous advantages in comparison with mechanical processes:

- Predominant fracturing along grain boundaries
- Liberation of morphologically intact minerals or micro-fossils
- Recovery of mono-mineral fragments
- Very clean surfaces of liberated minerals
- Minimal damage to liberated target specimens
- High yield of available target specimens
- Preservation of natural particle size distribution
- Narrow particle size distribution and choice of mean value
- Very low production of undesired fines below 50 µm
- No dust production
- Virtually no (cross-) contamination of samples

## Field of Application:

Selective fragmentation are used in following applications:

- Geoscience research
- Exploration of mining sites
- Analysis of composition of raw material
- Analysis of waste as secondary raw material

- E-scrap recycling
- Quartz glass recycling

#### **Operational Conditions and Limitations:**

The HV pulse generator is continuously charged by the HV power supply. When the predetermined voltage is reached, the energy of the HV pulse generator is discharged from the HV working electrode through the solid sample to the grounded bottom of the process vessel. This charging and discharging cycle repeats itself at a given frequency until the preselected number of pulses (discharges) has been reached.

Operational conditions and limitations for SelFrag are listed below.

Operational conditions

- Feed Rate: Up to 10 t/h (Pilot Plant)
- Largest single piece approx. 5 x 5 x 5 cm
- Voltage (output impulse generator) 90 200 kV
- Pulse frequency 1 5 Hz
- Working electrode gap 10 40 mm

## Limitations

- Particle size: 1 50 mm
- The achievable throughput depends on the average size of the particles and the comminution / liberation effect

**Development Status and Practical Experiences:** 

Innovative method; TRL 7 (Technology readiness levels)

Alternative Procedures:

Conventional comminution techniques

<u>Characteristics / Remarks:</u>With the selFrag-Lab the granular solid sample can be selectively fragmented into the different mono-mineral fractions with high yield and only small amounts of composite multi-mineral grains. For example granite, a typical crystalline granular rock, will be fragmented into the minerals quartz, feldspar and mica, which can be readily separated into pure mineral fractions.

References / Further Information:

Drzymala, J. (2007): Mineral Processing, Foundations of theory and practice of minerallurgy. Wroclaw: Oficyna Wydawnicza PWr.

Wang, E., Shi, F., Manlapig, E. (2005): Mineral liberation by high voltage pulses and conventional comminution with same specific energy levels. In: Minerals Engineering. SelFrag AG (2015): Application area, Geosciences, URL: http://www.selfrag.com/application-area.php [retrieved on 12.10.2015].

SelFrag AG (2006): selFrag Laboratory Fragmentator, URL: www.selfrag.com [retrieved on 09.10.2015].

# 3 Raw Material Recovery 3.2 Mineral Processing 3.2.4 Falcon Concentrator

## Author: Dr. Thomas Leißner

## **Objectives:**

The Falcon Laboratory Type Concentrator L40 is used for the enrichment of heavy minerals respectively the upgrading of low grade ores.

## <u>Abstract:</u>

Density separation is one of the oldest separation methods to beneficiate heavy minerals. It is based on the different movement of particles inside a fluid due to their size, shape and specific weight.

The two principals have to be distinguished which are settling and segregation. If settling is used, the separation is achieved by a difference in the settling speed of particles. This settling speed is related to the particle size and shape as well as the particle density. Besides the particle features the pulp density has an influence on the settling of particles. At low pulp densities there is free settling where neighboring particles do not affect the settling. At high pulp densities hindered settling appears. Particles can settle as clusters and zones. Furthermore fine particles where taken and moved by the fluid displaced by large particles while settling. When density separation by segregation e.g. in a flowing film is used the particles arrange in layers due their specific weight and size. Small and heavy particles move to the bottommost layer whereas light and large particles move to the above layers.

Separation in a Falcon Concentrator follows the principle of a flowing film.

Note: The size and shape of particles as well as their density have an influence on the settling speed. Therefor a classification into narrow size classes will enhance the quality of separation.

## Field of Application:

Separators working with a rotating bowl are used in upgrading of fine particles. Examples are recovery of fine gold in sand industry, pre-concentration of low grade ores before flotation, cleaning of flotation concentrates and heavy minerals beneficiation.

## **Operational Conditions and Limitations:**

Operational conditions and limitations for the Falcon laboratory type separator L40 used for beneficiation by density separation are listed below.

**Operational conditions** 

- Rotation: (10...80) Hz equals *g*-forces: ...300) *g*
- Throughput:
- Pulp: (5...30) vol.% solids
- Water pressure (standard bowl): (0.5...3) psi

Limitations

- Particle size: (5...500) µm
- Density: difference in density of valuables and gangue above 1 g/cm<sup>3</sup>

Development Status and Practical Experiences:

State of the art method.

#### Alternative Procedures:

Alternative procedures for density separation are:

- Shaking tables (slime tables)
- Jigs (coarse particles)

## Characteristics / Remarks:

Density separation is always influenced by the particle size and particle size distribution of the feed material. A classification into narrow size fractions before separation will lead to an improvement in the product quality.

#### References / Further Information:

Drzymala, J. (2007): Mineral Processing, Foundations of theory and practice of minerallurgy. Oficyna Wydawnicza PWr.

Schubert, H. (1996): Aufbereitung fester Stoffe – Sortierprozesse. Volume 2, 4th ed., Deutscher Verlag für Grundstoffindustrie, Stuttgart.

Weiss, N.L. (1985): SME Mineral Processing Handbook. Society of Mining Engineers of the American Institute of Mining, Metallurgical, and Petroleum Engineers.

Wills, B.A. (2006): Mineral Processing Technology. Butterworth-Heinemann.

## 3.2 Mineral Processing

3.2.5 Shaking Table

## Author: Dr. Thomas Leißner

## Objectives:

Shaking tables are used for the enrichment of heavy minerals respectively the upgrading of low grade ores.

## <u>Abstract:</u>

Density separation is one of the oldest separation methods to beneficiate heavy minerals. It is based on the different movement of particles inside a fluid due to their size, shape and specific weight.

The two principals have to be distinguished which are settling and segregation. If settling is used, the separation is achieved by a difference in the settling speed of particles. This settling speed is related to the particle size and shape as well as the particle density. Besides the particle features the pulp density has an influence on the settling of particles. At low pulp densities there is free settling where neighboring particles do not affect the settling. At high pulp densities hindered settling appears. Particles can settle as clusters and zones. Furthermore, fine particles where taken and moved by the fluid displaced by large particles while settling. When density separation by segregation e.g. in a flowing film is used the particles arrange in layers due their specific weight and size. Small and heavy particles move to the bottommost layer whereas light and large particles move to the above layers.

A shaking table can be described as a flat plate which swings linear in direction of the plate. Plates can be flat or with riffles of decreasing depth on their surface. The separation on the surface of a shaking table follows the principle of a flowing film. Due to the small film on top of the plate high throughputs just can be achieved by high pulp densities and large dimensions of the plate. As the latter is limited by principles of construction and the pulp density cannot be increased over around 30 vol.% solids, the capacity of shaking tables is limited to some tons an hour depending on particle size.

Further information concerning the design and principle of shaking can be found in the references.

Note: The size and shape of particles as well as their density have an influence on the settling speed. Therefor a classification into narrow size classes will enhance the quality of separation.

## Field of Application:

Shaking tables are used in upgrading of fine particles and slimes. They have been widely used in mineral processing in the past. An example is the beneficiation of heavy minerals like cassiterite.

## **Operational Conditions and Limitations:**

Operational conditions and limitations for shaking tables are listed below.

**Operational conditions** 

- side slope
- pulp: (15...30) vol.% solids (depending on the ore)
- amount of cleaning water
- amplitude and frequency of swinging

#### Limitations

- particle size: (40...3000) μm
- density: difference in density of valuables and gangue at least 1 g/cm<sup>3</sup>
- throughput (0.2 ... 5) t/h depending on particle size

**Development Status and Practical Experiences:** 

State of the art method.

#### Alternative Procedures:

Alternative procedures for density separation are:

- Separation in centrifuges (Falcon, Mozley, Knelson)
- Jigs (coarse particles)

## Characteristics / Remarks:

Density separation is always influenced by the particle size and particle size distribution of the feed material. A classification into narrow size fractions before separation will lead to an improvement in the product quality.

#### References / Further Information:

Drzymala, J. (2007): Mineral Processing, Foundations of theory and practice of minerallurgy. Oficyna Wydawnicza PWr.

Schubert, H. (1996): Aufbereitung fester Stoffe – Sortierprozesse. Volume 2, 4th ed., Deutscher Verlag für Grundstoffindustrie, Stuttgart.

Weiss, N.L. (1985): SME Mineral Processing Handbook. Society of Mining Engineers of the American Institute of Mining, Metallurgical, and Petroleum Engineers.

Wills, B.A. (2006): Mineral Processing Technology. Butterworth-Heinemann.

## 3.2 Mineral Processing

3.2.6 Hydrocyclone

## Author: Dr. Thomas Leißner

## **Objectives:**

Classification of fine particles prior to physical or physicochemical separation.

## Abstract:

Hydrocyclones are used for wet classification of fine particles. Classification is needed to improve selectivity of downstream separation processes which leads to higher grades and higher recovery. Fine particles tend to unselectively report to tailings or concentrate due to particle interactions and entrainment. Furthermore, most of separation processes are affected by particle size. That is why narrow size classes or a feed free of fines are favored in separation.

Hydrocyclones can also be used as classifier in grinding circuits to split of particles in product fineness from ones still to coarse. The coarse fraction is send back to the mill for regrinding.

The working principle can be described as follows: The pulp is forced on a downwards directed rotational flow by the design of the inlet nozzle. This leads to centrifugal forces causing sedimentation in radial direction. When the pulp flows in the conical part of the cyclone, the fluid is split into a part flowing upwards and leaving the cyclone through the vortex finder (fines) and a part leaving through the spigot (apex orifice, coarse fraction).

Picture showing a cyclone and its important dimensions as well as formulas.



mineral processing, industrial minerals, process engineering

**Operational Conditions and Limitations:** 

#### Sample

- particle size  $(x_{50})$  and shape (isometric, plates, fibers)
- solid content in feed: (10...15) vol.% (good efficiency), (15...30) vol.% (deteriorating efficiency)
- cut size: (10…100) μm

## Cyclone

- diameters: cyclone  $d_{c}$ , inlet  $d_{i}$ , apex  $d_{a}$ , vortex  $d_{v}$ , (sketch)
- throughput, pressure

## **Development Status and Practical Experiences:**

State of the art technology.

Alternative Procedures:

Wet screening down to 40 µm cut size.

Characteristics / Remarks:

It has to be taken into account, that settling speed is a function of particle size, particle shape and density. Light coarse and heavy fines will therefor report to the same product.

References / Further Information:

Schubert, H. (1989): Aufbereitung fester mineralischer Rohstoffe. Volume 1, 4th ed., Deutscher Verlag für Grundstoffindustrie, Leipzig.

Weiss, N.L. (1985): SME Mineral Processing Handbook. Society of Mining Engineers of the American Institute of Mining, Metallurgical, and Petroleum Engineers.

Napier-Munn, T., Wills, B.A. (2006): Will's Mineral Processing Technology. 7th ed., Butterworth-Heinemann.

## 3.2 Mineral Processing

3.2.7 Wet magnetic separation

Authors: Thomas Felbinger, Dr. Sebastian Prinz

## Objectives:

By creating an environment comprising a magnetic force (Fm), a gravitational force (Fg) and a drag force (Fd) magnetic particles can be separated from nonmagnetic particles.

## <u>Abstract:</u>

For wet high intensity magnetic separation a magnetic field is generated via a water cooled electromagnet fixed to the processing unit. The processing chamber contains flux converging metal elements (matrix). These elements have sharp edges to which the suspended feed material is exposed. Each edge becomes highly induced during operation and provides the necessary high-intensity, high-gradient field to separate or attract weakly magnetic particles. Magnetic particles are collected in the matrix during operation while nonmagnetic particles pass through the magnetic zone. After having separated the material, magnetic particles are flushed out by feeding water through the pole box while the magnet is turned off.

A wet high intensity magnetic separator manufactured by Eriez Magnetics was used to run the tests. The nominal maximum magnetic field of the magnetic separator is 2 Tesla.

## Field of Application:

Wet magnetic separation is used in upgrading of fine particles. They have been widely used in mineral processing in the past. An example is the beneficiation of kaolinite (brightening); Fe2O3 reduction in glass sand, feldspar and barite; Cu-reduction in Mo concentrates; deashing and desulphurization of coal; phosphates upgrading.

**Operational Conditions and Limitations:** 

Operational conditions and limitations for wet magnetic separation are listed below.

**Operational conditions** 

- Magnetic field in separation zone (on matrix surface) 2 20 kGauss
- Solids in feed: as high as possible. For clays limited to about 30% solids by weight due to viscosity problems.
- Typical cycle times (Kaolin separation): 4 min.

## Limitations

- Particle size 20 - 1000 µm (Oversize particles will block the matrix)

**Development Status and Practical Experiences:** 

State of the art method

Characteristics / Remarks:

Matrix design: the electromagnetic field is disturbed by introducing a ferromagnetic material with sharp edges and high magnetic field gradients are created. These disturbing elements (filaments) are spaced apart to allow pulp flow around them.

Drzymala, J. (2007): Mineral Processing, Foundations of theory and practice of minerallurgy. Wroclaw: Oficyna Wydawnicza PWr.

Schubert, H. (1977): Aufbereitung fester mineralischer Rohstoffe – Sortierprozesse. Volume 2, 2nd ed., Deutscher Verlag für Grundstoffindustrie, Leipzig.

Metso Corporation (2015): Basics in Minerals Processing. 8th ed.

3 Raw Material Recovery				
3.2 Mineral Processing	3.2.8	High Separatio	Gradient on	Magnetic

Authors: Thomas Felbinger, Dr. Sebastian Prinz

## **Objectives:**

By creating an environment comprising a magnetic force (Fm), a gravitational force (Fg) and a drag force (Fd) magnetic particles such as heavy minerals and steal wear can be separated from nonmagnetic particles.

## Abstract:

A particle placed in a magnetic field interacts with this field. As a result, the particle moves in the field. This phenomenon is utilized in separation of particles of different materials and it is termed magnetic separation. The utilized feature of the material is magnetic susceptibility. For particles pulled into stronger magnetic fields magnetic susceptibility is positive (paramagnetics) and for those which are pushed out is negative (diamagnetics). Magnetic separation is possible for particles of different signs or susceptibilities.

Most heavy minerals have paramagnetic or even ferromagnetic properties. Therefore a magnetic force acts on these minerals with increasing magnetic field strength. Since this force is strong in case of ferromagnetic minerals only moderate magnetic field strengths are necessary for separation. To separate paramagnetic minerals, higher field strengths need to be applied.

#### Field of Application:

High gradient magnetic separation has been widely used in mineral processing in the past for upgrading a wide variety of different ores. An example is the removal of paramagnetic minerals (biotite, muscovite, chromite, columbite-tantalite, ilmenite, etc) and fine, weakly magnetic particles from a range of non-metallic industrial minerals, such as:

- silica sand for glass production
- feldspar for ceramics
- beach sands
- silicon carbides
- magnesites
- other dry industrial minerals

#### **Operational Conditions and Limitations:**

Operational conditions and limitations for high force magnetic separation are listed below.

**Operational conditions** 

- Magnetic field in separation zone (on matrix surface) 2 – 20 kGauss

Limitations

- Particle size 75 µm - 13 mm

Development Status and Practical Experiences: State of the art method.

<u>Alternative Procedures:</u>Alternative procedures for high gradient magnetic separation are:

- Froth Flotation
- <u>Electrostatic separation</u>

## Characteristics / Remarks:

For a successful separation of minerals their magnetic susceptibility must be significantly different. According to Dobby et al. (1979) the ratio of magnetic susceptibility of particles undergoing separation should be at least as 20 to 1. The selectivity of separation depends not only on the differences in magnetic susceptibility but also on the changes of the susceptibility with magnetic field, as well as on particles size. Therefore, each raw material can be subjected only to a carefully chosen type of separator.

## References / Further Information:

Drzymala, J. (2007): Mineral Processing, Foundations of theory and practice of minerallurgy. Wroclaw: Oficyna Wydawnicza PWr.

Schubert, H. (1977): Aufbereitung fester mineralischer Rohstoffe – Sortierprozesse. Volume 2, 2nd ed., Deutscher Verlag für Grundstoffindustrie, Leipzig.

Metso Corporation (2015): Basics in Minerals Processing. 8th ed.

## 3.2 Mineral Processing

3.2.9 Sensor based sorting

Authors: Thomas Felbinger, Dr. Sebastian Prinz

## Objectives:

Sensor-based sorting, is an umbrella term for all applications where particles are singularly detected by a sensor technique and rejected by an amplified mechanical, hydraulic or pneumatic process. Modern, automated sorting applies sensors (visible spectrum, near infrared, X-ray, ultraviolet), that can be coupled with electrical conductivity and magnetic susceptibility sensors, to control the mechanical separation of ore into two or more categories on an individual rock by rock basis. Also new sensors have been developed which exploit material properties such as electrical conductivity, magnetization, molecular structure and thermal conductivity. The technique is generally applied in the three industries mining, recycling and food processing. Sensor based sorting has found application in the processing of a wide range of different ores and raw materials including nickel, quartz, salt, gold, copper, coal and diamonds.

## Abstract:

Sensor based sorting devices use characteristics of individual particles for sorting of minerals, e.g. color, chemical composition, contrast or shape. Before sorting, the raw material is discharged to a vibration feeder which allows a constant feeding speed and adjusts a homogenous distribution of the feed material (mono layer). The vibration feeder transports the sample to the scanning line, where a detection system scans the material. The information from the detection system is evaluated by a computer system and then provided to the high pressure air nozzle ejection system, which separates in-specified and off-specified particles by application of high pressure air jets. A schematic diagram of the working principle of a sensor based sorter is illustrated in the Figure.



Figure: Schematic diagram of sensor based sorting machine

## Field of Application:

Sensor based sorting is generally applied in mining, recycling and food processing and used in the particle size range between 1 and 300 mm. Applications in the mining sector are:

- Magnesite, Calcite, Talc, Gold, Quartz, Phosphate, Borate, Feldspar, Burnt, Lime, Coal, Base metals, Calcium Carbonates, Phosphate, Barite, Salt

Operational conditions and limitations for sensor based sorting are listed below.

**Operational conditions** 

- Feed Rate: Up to 150 t/h
- Sensor Configurations which can be equipped: Color, Near-Infrared (NIR), X Ray Fluorescence (XRF), X Ray Transmission (XRT), high sensitive Electromagnetic (EM) sensor

#### Limitations

- Particle size: 1 300 mm
- The achievable throughput depends on the average size of the particles and the number of particles to be rejected

**Development Status and Practical Experiences:** 

State of the art method.

#### Alternative Procedures:

Alternative procedures for sensor based sorting are:

• Optical sensor: hand sorting

#### Characteristics / Remarks:

The configurations of the optical sorting process depend on different parameters:

- There must be a visible difference in the color or brightness of the materials to be separated
- The material is fed to the sorting process in narrow particle size ranges. The sorting parameters or air pressure can then be optimally adjusted to the specific fraction
- The material must allow for liberation of individual, i.e. single particles

References / Further Information:

Pretz, T., Wotruba, H. (2012): Sensor Based Sorting 2012, Heft 128 der Schriftenreihe der GDMB Gesellschaft für Bergbau, Metallurgie, Rohstoff- und Umwelttechnik. Clausthal-Zellerfeld, GDMB-Informationsgesellschaft mbH.

TOMRA Systems ASA (2015): Mining Technology, https://www.tomra.com/en/solutionsand-products/sorting-solutions/mining/mining-technology/ [retrieved on 12.10.2015].

Dehler, M. (2003): Optical Sorting of Mineral Raw Materials. AUFBEREITUNG TECHNIK 44, Nr. 10, 38-42

Bergmann, J.-M. (2011): Sensor-based sorting. Industrial Minerals, July 2011 issue, 58-62.

3 Raw Material Recovery	

#### 3.2 Mineral Processing

3.2.10 Flotation

Authors: Thomas Felbinger, Dr. Sebastian Prinz

## Objectives:

Flotation is a mineral separation process, which takes place in water-mineral suspension. Froth flotation is a highly versatile method for separating particles based on differences in the ability of air bubbles to selectively attach to the surface of specific minerals. Froth flotation is a highly selective concentration process.

## Abstract:

Flotation is a wet mineral separation process. Froth flotation is used for selectively separating minerals by taking advantage of differences in their hydrophobicity. The surface of selected minerals is made hydrophobic (water-repellent) by conditioning with suitable and highly selective reagents. The hydrophobic particles become then attached to air bubbles that are introduced into the suspension and are carried to a froth layer above the liquid, thereby being separated from the hydrophilic (wetted) particles. Flotation process designs vary in complexity depending primarily on the type of mineral, degree of liberation and the desired quality of the product.

Flotation is normally undertaken in several stages to maximize the recovery of the target mineral or minerals and the concentration of those minerals in the concentrate, while minimizing the energy input.

## Field of Application:

A wide variety of different ores and raw materials can be upgraded by flotation. Since ores contain minerals of different properties, a highly selective collector is needed for efficient flotation of one or a group of minerals. The flotation process is used for the separation of a large range of sulfides, carbonates and oxides prior to further refinement. Phosphates, graphite and coal are also upgraded (purified) by flotation technology.

## **Operational Conditions and Limitations:**

Operational conditions and limitations for flotation are listed below.

**Operational conditions** 

- Solids content: 4 50 wt.-% solids (depending on the ore)
- Retention time 4 20 min (depending on the ore)
- Cell size 3 200 m<sup>3</sup> (Reactor cell flotation system (RCS))

## Limitations

- Feed particle size is typically less than 0.3 mm
- Hydrophobicity of the minerals to be separated is a necessary prerequisite for direct flotation
- throughput depending on retention time

**Development Status and Practical Experiences:** 

State of the art method.

#### Alternative Procedures:

Froth flotation can in some instances be replaced (especially in arid environmental conditions) by electrostatic separation which is also a surface sensitive method exploiting differences in surface charge

#### Characteristics / Remarks:

In addition to the reagents added, the flotation process depends on following main parameters:

- Hydrophobicity: The main material parameter of the flotation is hydrophobicity.
- **Liberation**: the ore to be treated is reduced to fine particles by crushing and grinding so that the various minerals are present as physically separate grains.
- **Retention time**: needed for the separation process to be effective; determines the volume and number of flotation cells required
- **Agitation** and **aeration**: needed for optimum flotation conditions, determine the types of flotation mechanism and the power input required.

## References / Further Information:

Drzymala, J. (2007): Mineral Processing, Foundations of theory and practice of minerallurgy. Wroclaw: Oficyna Wydawnicza PWr.

Schubert, H. (1977): Aufbereitung fester mineralischer Rohstoffe – Sortierprozesse. Volume 2, 2nd ed., Deutscher Verlag für Grundstoffindustrie, Leipzig.

Fuerstenau, M., Jameson, G., Yoon, R. (2007): Froth Flotation, A Century of Innovation. Colorado: Society for Mining, Metallurgy, and Exploration, Inc. (SME).

Metso Corporation (2015): Basics in Minerals Processing. 8th ed.

Pease, J.(2007): Increasing the energy efficiency of grinding Presented at: Crushing and Grinding, Brisbane, September 2007.

3.3 Hydrometallurgy

3.3.1 Acid Digestion

## Author: Dr. Carsten Pätzold

## **Objectives:**

Acid digestion of sulfide-rich dump material for metal recovery

## Abstract / Method:

Fine-grained flotation tailings from former metal enrichment processes contained in dump material represent a potential source for metal recovery. Generally, sulfides like sphalerite (ZnS) and galena (PbS) are present beside prevalently occurring Pyrite and Marcasite (FeS<sub>2</sub>). However, concentration in these residues is often low (<1.0 wt.%) for which reason modern flotation steps and further separation techniques have to be carried out leading to concentrates preferred as starting material for chemical treatment. Presence of valuable elements should be considered especially during this enrichment process.

Acid digestion is a promising method for sulfide-rich dump material. Tests with mineral acids as well as other chemicals show that nitric acid is particularly advantageous for a high yield rate bringing the compounds in a soluble form. It is sufficient to use a half-concentrated HNO<sub>3</sub>, what is relevant under consideration of the economic efficiency.

Treatment with HNO<sub>3</sub> can be performed without any heating at normal pressure with dried, but also wet material. Advantageous is a time of 5 hours at an acid/dump material weight ratio of 4:1. Nitrates, but also PbSO<sub>4</sub> are formed and can be separated by filtration. Beforehand, Pb(NO<sub>3</sub>)<sub>2</sub> formed should be precipitated by addition of Na<sub>2</sub>SO<sub>4</sub>. Solid residue can be used for recovery of Sn, Pb or other metals for example. The filtrate is preferably extracted with MTBE for acid recovery. Recuperated HNO<sub>3</sub> is passed back into the cycle and can be reused for digestion.



Figure: Flow sheet of acid digestion of sulfide-rich dump material

Depending on the composition, further steps for metal recovery are necessary. *Examples:* 

- Precipitation of Fe as jarosite
- Precipitation of FeAsO<sub>4</sub>
- Electrolysis
During acid digestion of sulfide-rich dump material formation of hydrogen sulfide and sulphur has to be taken into account.

# Field of Application:

Lower concentrated, solid dump materials

Residues from former mining activities, especially sulfide-rich dump material

**Operational Conditions and Limitations:** 

**Operational Conditions:** 

Recovery of metals from sulfide-rich dump materials

Limitations:

Because of the low metal content, a concentration step is necessary for enrichment prior to the recovery process.

Development Status and Practical Experiences:

Laboratory tests were successfully completed with concentrates from sulfide-rich dump material.

Alternative Procedures:

Roasting process  $\rightarrow$  pyrometallurgical process

Characteristics / Remarks:

Characteristics:

Digestion process at low temperature and normal pressure; possibility of reusing of unspent acid

Remarks:

Price-intensive is the used mineral acid therefore a concentrate should be produced prior to the recovery process for increasing the metal content/acid ratio.

References / Further Information:

Freiberg University of Mining and Technology Institute of Chemical Technology Leipziger Str. 29 D-09599 Freiberg / Germany

## **3 Raw Material Recovery**

## 3.3 Hydrometallurgy

3.3.2 Bioleaching

## Author: Mirko Martin

#### **Objectives:**

Bioleaching aims at solubilization of ore minerals for subsequent metal recovery. Bioleaching is restricted to minerals that can be attacked by microbiological processes. Therefore, mainly sulfidic ores can be bioleached.

## Abstract:

Preliminary experiments to determine suitable conditions (pH, Temperature, residence time) were conducted in shaking flasks. After A mixed culture of e.g. acidophilic sulfide and iron oxidizing bacteria was used for bioleaching of flotation tailings (suitable grain size < 63  $\mu$ m) in a batch bioreactor system at the required pH under ambient conditions.

In the experiments maximum pulp densities of 40% (w/v) were used with simultaneous recovery of 76% Zink and 84% Indium. Using a lower solid content higher recovery rates for Zink of up to 100% could be achieved. The metal concentration in the PLS is lower so that a thorough optimization is required. Even without inoculation with adapted microorganisms the bioleaching was successful, however, bioleaching with autochthonous microorganisms in the tailings material was slower.

By addition of Fe<sup>2+</sup> to the medium (100 mM) the bioleaching process was slightly accelerated. Without adjustment of the pH by acid addition bioleaching was slower but similar metal recovery for Zink and Indium could be achieved. The optimized process conditions can be applied in a pilot scale tank leaching operation for leaching of the flotation tailings material. The limited supply of nutrients without further additives minimizes the OPEX costs for the process. A heap bioleaching procedure is not recommended due to the low grain size of the material.

#### Field of Application:

Tailings containing sulfidic ores containing sphalerite, chalcopyrite, sphalerite

**Operational Conditions and Limitations:** 

restricted mainly to sulfidic ore

- Operation in bioreactor containing water and basal salts for supply with nitrogen
- pH adjustment by H<sub>2</sub>SO<sub>4</sub> or NaOH, respectively
- inoculation with iron and sulfur oxidizing bacteria (10<sup>7</sup> cell/ml)
- room temperature
- maximum pulp density: 40 (w/v) %

# Results:

10 % solids: Zn: 81%, In: 87%

40 % solids: Zn: 76%, In: 84%



Figures: Recovery rate of As, Cd, Fe, In and Zn from tailings over a leaching time of 25 days

# **Development Status and Practical Experiences:**

The process was developed at laboratory scale (batch scale experiments in shake flasks, columns and bioreactor) for sulfidic flotation tailings.

Upscaling to pilot plant scale is in progress



# Alternative Procedures:

Conventional processing of fine grained tailings is done by gravity separation and flotation. Alternative procedures are chemical Leaching (e.g. acid leaching, pressure leaching) and pyrometallurgy.

# Characteristics / Remarks:

In case of the investigated sulfidic Zn/Pb tailings with high amounts of pyrite/marcasite no pH adjustment by acid addition was not necessary for a high metal recovery but accelerated the bioleaching process.

# References / Further Information:

Martin, M., Janneck, E., Kermer, R., Patzig, A., Reichel, S. (2015): Recovery of indium from sphalerite ore and flotation tailings by bioleaching and subsequent precipitation processes. Minerals Engineering, 75, 94-99.

3 Raw Material Recovery	
3.3 Hydrometallurgy	3.3.3 Hydrometallurgical Processing for Pregnant Leach Solutions (PLS)

#### Author: Mirko Martin

**Objectives:** 

- Hydrometallurgical processing of PLS aims at
- Recovery of valuable metal contents from PLS in an economic way
- concentrate the metals in a concentrate ore produce directly salable metals

## Abstract:

The PLS resulting from bioleaching of tailings containing residual base metal ores are characterized by

- high amounts of iron and sulphate
- variable amounts of valuable metals
- variable amounts of hazardous components like cadmium or arsenic

For processing of the PLS from tailings of sulfidic Pb/Zn ores containing indium, a technology comprising stepwise precipitation was applied.

In a first step precipitation was done by slightly increasing the pH to generate a precipitate of Fe hydroxides containing the In and the As.

In a second step the Zn content is precipitated by adjusting the pH to  $\approx$ 10 as Hydroxide. Alternatively Zn may be recovered by electrolysis after purification of the solution.

The precipitate of step 1 is dissolved in  $H_2SO_4$ . In the solution Fe(III) is reduced to Fe(II) e.g. by  $SO_2$ . From the resulting solution the In is recovered by liquid extraction with DE2HPA. The organic phase is stripped by  $H_2SO_4$  and the In is precipitated by addition of NaOH as Hydroxide. Alternatively the In may be recovered by electrolysis of the stripping solution.

Hazardous components are concentrated in iron compounds (As) or are precipitated from resulting solutions (Cd), respectively.

#### Field of Application:

Developed technology is applicable for metal recovery from leaching solutions, especially from bioleaching. It takes into account rare metals, especially indium.

# **Operational Conditions and Limitations:**

1. Precipitation process

- Application of a stepwise precipitation process
- Stepwise precipitation enables Indium separation from AI, most of ferric iron and divalent ions (Zn, Cd, Cu) (are not precipitated at low pH)

In the first step a pre-concentrate of trivalent metals is precipitated by slightly increasing pH of the solution. Precipitation of metals like indium is enhanced by the arsenic content of the solution.



Precipitation of divalent metals (e.g. Zn) is subsequently achieved by pH increase >7. Further processing options comprise liquid extraction of Zn or Cu and electrowinning from the stripping solutions.

2. Solvent extraction process

By combining precipitation and solvent extraction processes a more profitable operation is possible.

The pre-concentrate of the first step is dissolved in  $H_2SO_4$ . In the solution Fe(III) is reduced to Fe(II) e.g. by SO<sub>2</sub>. From the resulting solution the In is precipitated as Hydroxide after purification. The Hydroxide is decomposed to  $In_2O_3$ .

In an alternative processing route the In is recovered by liquid extraction with DE2HPA after pH adjusting. The organic phase is stripped by  $H_2SO_4$  and the In is precipitated by addition of NaOH as Hydroxide. Alternatively the In may be directly recovered by electrolysis of the stripping solution.

Indium hydroxide or oxide may be further processed pyrometallurgy.

**Development Status and Practical Experiences:** 

The process was developed in laboratory scale for PLS from bioleaching of sulfidic Zn/Pb ore flotation tailings containing indium.

Alternative Procedures:

Conventional processing of fine grained tailings is done by gravity separation and flotation. Alternative procedures are chemical Leaching (e.g. acid leaching, pressure leaching) and pyrometallurgy.

Characteristics / Remarks:

References / Further Information:

Martin, M., Janneck, E., Kermer, R., Patzig, A., Reichel, S. (2015): Recovery of indium from sphalerite ore and flotation tailings by bioleaching and subsequent precipitation processes. Minerals Engineering, 75, 94-99.

# **3 Raw Material Recovery**

# 3.4 Pyrometallurgy

## Author: David Algermissen

## **Objectives:**

Pyrometallurgic treatment of reduction agents for the maximum recovery of valuable metal oxides.

# Method description:

Several tests were performed with selected materials from the Thuringia model dump using melting furnace systems at the laboratory of the FEhS institute: several kilograms of the material were heated in a Tammann furnace up to a temperature of approx. 1650 °C in reducing atmosphere. Special consideration had to be given to the carbon monoxide/carbon dioxide gases released by the redox reduction of metal oxides with carbon in the melting liquid phase. The gas released from the melt leads to foaming phenomenon which requires a special geometric form of the melting crucible or the limitation of the added amount, i.e. so that in each step only a small amount would be reduced. For the tests in this project, the discontinuous addition was chosen, which facilitates the reduction of slag in the crucible during a hold time before new material is being fed in again. The total melt testing time in the laboratory amounted to about 17 hours per trial. This, however, does not allow any conclusions on the required treatment time of a large-scale technical process.

## Field of Application:

The pyrometallurgical treatment of the materials in the lab allows the simulation of normal operation facilities such as shaft furnaces so that all materials with metal oxide fractions can be recovered at the ordinary operation temperatures. The use of different reduction agents such as carbon or aluminum is possible.

**Operational Conditions and Limitations:** 

The treatable amount is limited depending on the amount of crucible material used. While roughly 2 kg may be produced by the carbon reduction, the amount involving other reduction agents is limited to about 250 gr, since specific crucibles with limited volumes are needed.

#### **Development Status and Practical Experiences:**

Well established state of technology in the lab of the FEhS institute. The crucibles specifically manufactured for the melting with reduction agents other than carbon represent an advantage (unique selling point) of the FEhS that has been used for this project.

# Alternative Procedures:

In particular, for the reduction with reduction agents other than carbon, there are no known alternatives owing to the unique characteristics/properties of the crucibles.

# References / Further Information:

Contact: mineralische-rohstoffe@bgr.de ISBN: 978-3-948532-58-1 (PDF) DOI: https://doi.org/10.24406/umsicht-n-645892