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GTK

MINE WASTE CHARACTERIZATION: COMMON AND NOVEL METHODS

Morecovery Educational Workshop, May 5th 2020

Teemu Karlsson

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ENVIRONMENTAL IMPACTS

- Mines can generate acid rock drainage (ARD) or neutral rock drainage (NRD) containing harmful substances, which is one of the main environmental issues of the mining industry
- This particularly applies to deposits containing sulphide minerals, which are prone to oxidization under the influence of atmospheric conditions
- Drainage quality largely depends on the mineralogical and chemical composition of the mine wastes, and particularly on the ratio of acid-producing and neutralizing minerals, combined with reactions catalysed by microbes
- Mine waste facilities, e.g. waste rock piles and tailings impoundments, are the main sources of harmful drainage

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CHARACTERIZATION, WHY?

- The characterization is needed for the prediction, prevention and management of the environmental impacts of the mining wastes
- The prediction of the mining wastes potential to produce ARD and other harmful drainage is crucial when planning the waste facilities, and estimating the utilization possibilities of the waste materials and selecting suitable methods for the closure and rehabilitation of the waste facilities
- Thus it is important to characterise mine wastes and assess their behaviour before actual mining activities begin!

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METHODS IN GENERAL

- The short- and long-term behaviour of mine wastes can be predicted e.g. by:
  - *Static tests*
  - *Kinetic tests*
  - *Leaching tests*
  - *Mineralogy*
  - *Geochemical modelling*
  - *Analogies from similar older mine waste sites*

Method	Description	Reference / Standard / Guide
Mineralogical methods		
Optical microscopy (OM)	Transmitted/reflected light microscopy, point-counting of minerals	EN 932-3:1996 and amendment A1:2003 (SFS 2003)
Scanning electron microscopy (SEM)	Information on e.g. mineral abundances, chemical composition, grain size and microstructures	e.g. Swapp 2014
X-ray diffraction (XRD)	Quantitative and qualitative mineralogy for crystalline samples, especially clay minerals	e.g. Reynolds 1989
Particle size measurements		
Sieving	PSD for particles >0.063 mm	SFS-EN 933-1 (SFS 2012)
Wet method laser diffraction	PSD for particles <0.063 mm, based on scattering of a laser beam	e.g. Cepuritis et al. 2017
X-ray sedimentation	PSD for particles <0.063 mm, based on particle sedimentation speed and equivalent Stokes diameter	e.g. Cepuritis et al. 2017
Static tests and related determinations		
Modified ABA test	Quantifying the potential of a sample to produce and neutralize acid	CEN/EN 15875 (CEN 2011), Sobek et al. 1978
Total sulphur for acid production potential (AP)	S combustion in e.g. a LECO furnace	ISO 15178 (ISO 2000)
Neutralisation potential (NP)	Titration by HCl to pH 2.0–2.5	CEN/EN 15875 (CEN 2011), Lawrence & Wang 1997
Net acid generation (NAG)	Acid generation based on the reaction of a sample with hydrogen peroxide	Miller et al. 1990, AMIRA 2002
Chemical extractions		
XRF	Total element concentration	Criss & Birks 1968
Aqua regia	Leaching of sulphide fraction	ISO 11466 (ISO 1995), Doležal et al. 1968, Heikkinen & Räisänen 2009
Ammonium oxalate	Leaching of Fe(III)oxyhydroxides, Fe(III)oxides	Räisänen et al. 1992, Dold 2003, Heikkinen & Räisänen 2008
Ammonium acetate	Leaching of exchangeable and carbonate fractions	Gatehouse et al. 1977, Sondag 1981, Dold 2003, Heikkinen & Räisänen 2008
Ammonium chloride	Leaching of physically adsorbed, easily leachable, bioavailable fractions	Heikkinen & Räisänen 2008
Leaching tests		
Two-stage batch leaching test	Leaching of water-soluble fraction	CEN/EN 12457-3
Column leaching test	Leaching of water-soluble fraction	CEN/TS 14405 (CEN 2017)
Kinetic tests		
Humidity cell test (HCT)	Small-medium scale laboratory test	ASTM 2013, CEN/TR 16363 (CEN 2012)
Field scale lysimeter test	Non-standardized medium- to large-scale field test	Hansen et al. 2000
Large-scale test pile	Non-standardized large-scale field tests	INAP 2009, MEND 2012

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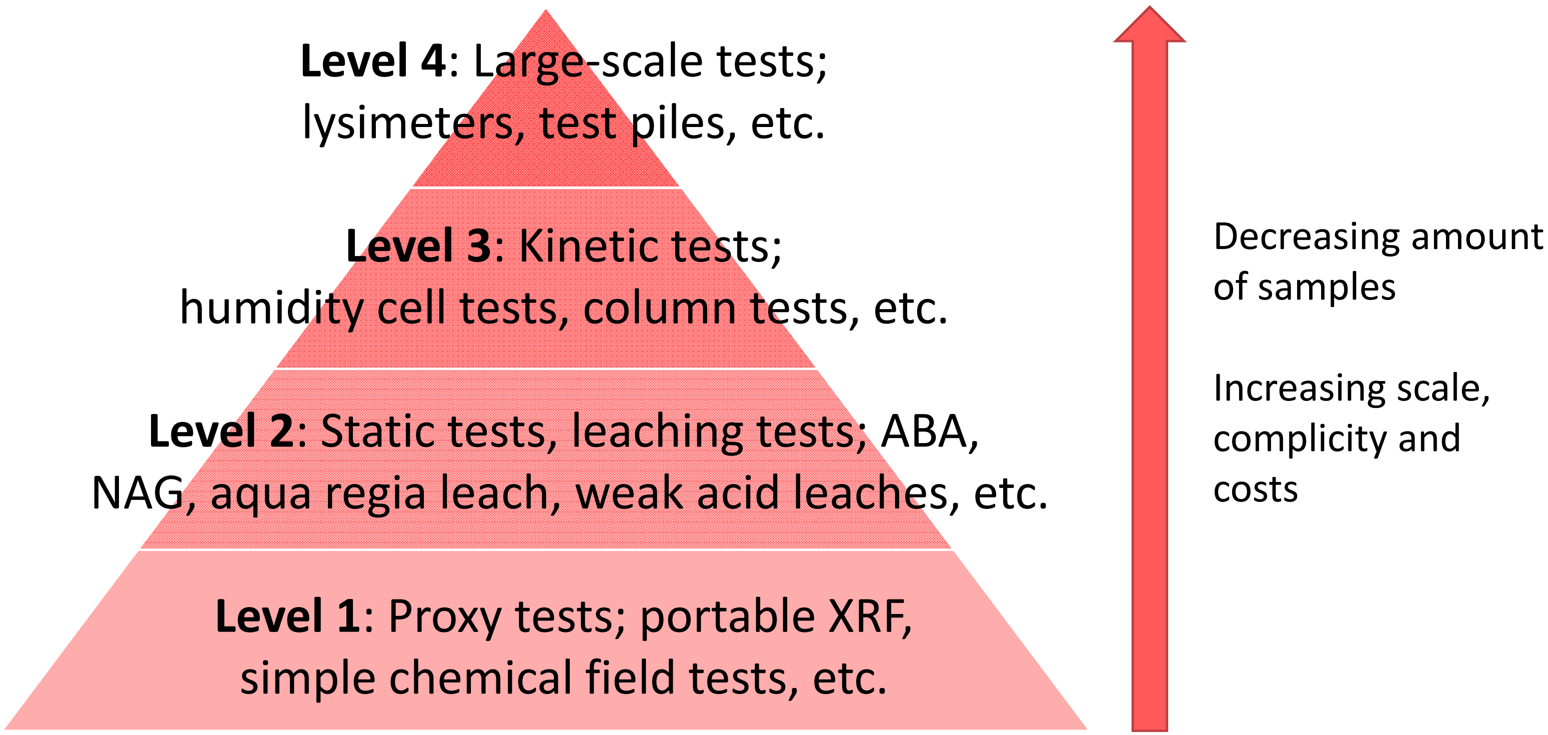
METHODS IN GENERAL



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EUROPEAN LEGISLATION CONCERNING MINE WASTE CHARACTERIZATION

- COMMISSION DECISION of 30 April 2009 completing the technical requirements for waste characterisation laid down by Directive 2006/21/EC of the European Parliament and of the Council on the management of waste from extractive industries (2009/360/EC)
- Technical Committee CEN/TC 292 2012. Characterization of waste – Overall guidance document for characterization of wastes from extractive industries. CEN/TR 16376:2012
- European Commission 2009. Reference Document on Best Available Techniques for Management of Tailings and Waste-Rock in Mining Activities. January 2009

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SAMPLING

- Representative sampling is the key element in successful characterization
- Correct environmental sampling techniques are presented, for example, by:
  - *McLemore et. al (2014) Sampling and monitoring for the mine life cycle*
  - *Sädbom & Bäckström (2018) Sampling of mining waste – historical background, experiences and suggested methods*

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PREDICTION OF ACID DRAINAGE

- The acid production potential of mine waste is usually determined based on different acid-base accounting (ABA) tests
  - *The standardised method SFS-EN 15875 being widely used in Europe*
- Another commonly used method is the single-addition net acid generation (NAG) test
  - *Involves the reaction of a sample with hydrogen peroxide to rapidly oxidise any sulfide minerals present*
  - *For the method see Miller et al. 1997 and AMIRA 2002*

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PREDICTION OF ACID DRAINAGE

- The widely used static ABA and NAG tests have known limitations related to the mineralogy of the sample material, for example:
  - *Acid production potential (APP) may be overestimated if there are other sulphide or sulphur-containing minerals than pyrite*
  - *Conversely, the APP may be underestimated if the waste contains large amounts of easily dissolvable and acid-generating iron sulphate minerals or siderite*
  - *The NP may be underestimated if the weathering of silicate minerals is not considered in APP estimations*
  - *Static tests do not indicate the source minerals of the NP and AP*
  - *See e.g. White III et al. 1999; Paktunc 1999; Jambor 2003; Parbhakar-Fox and Lottermoser 2015; Dold 2017; Parbhakar-Fox et al. 2018*

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PREDICTION OF ACID DRAINAGE

- To overcome the limitations, some mineralogical-based approaches have been proposed
  - *The ARD potential can also be calculated based on the mineralogy of the sample (Dold 2017; Karlsson et al. 2018b)*
  - *See also: Lawrence and Scheske (1997), Parbhakar-Fox and Lottermoser (2015) and Jamieson et al. (2015)*

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PREDICTION OF ACID DRAINAGE

- Relative reactivity in terms of the acid-neutralization capacity for selected minerals after Sverdrup (1990) and Kwong (1993)

Mineral class	Typical minerals	Relative reactivity[1)]			
		Average mineral class content			
		100%	30%	3%	0.3%
Carbonates	Calcite, dolomite, magnesite, aragonite, brucite	1	1	1	1
Fast weathering	Anorthite, olivine, garnet, diopside, wollastonite, jadeite, nepheline, leucite, spodumene	0.6	0.67	0.3	0.1
Intermediate weathering	Enstatite, augite, hornblende, tremolite, actinolite, biotite, chlorite, serpentine, talc, epidote, zoisite, hedenbergite, glaucophane, anthophyllite	0.4	0.2	0.03	0.01
Slow weathering	Plagioclase (Ab100-Ab30), kaolinite, vermiculite, montmorillonite, gibbsite	0.02	0.013	0.002	-
Very slow weathering	K-feldspar, muscovite	0.01	0.007	0.001	-
Inert	Quartz, rutile, zircon	0.004	0.0007	-	-

- More research needed to define more accurate relative reactivities

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PREDICTION OF ACID DRAINAGE

- Example calculation for the mineralogical APP, NNP and NPR (Karlsson et al. 2018b)

Mineral	% total mass		Mineral	wt. % S in mineral	wt. % of mineral in sample	wt. % S in sample	minAP1 (31.25)	minAP2 (62.5)
Plagioclase	35.0		Pyrrhotite	37.67[1)]	0.50	0.19	2.94[2)]	5.89
Quartz	25.0		Pyrite	53.30	0.30	0.16	5.00[3)]	9.99
Biotite	20.0		Total minAP				7.94	15.88
Mg-Hornblende	6.0			wt. % C in mineral	wt. % of mineral in sample	wt. % C in sample	minNP	
K-feldspar	6.0		Calcite	12.00	0.20	0.02	1.67[4)]	
Fe-Hornblende	4.0		Biotite		20.00		61.94[5)]	
Serpentine	3.0		Mg-Hornblende		6.00		4.90[6)]	
Pyrrhotite	0.5		Fe-Hornblende		4.00		2.83[7)]	
Pyrite	0.3		Serpentine		3.00		6.69[8)]	
Calcite	0.2		Total minNP				78.03	
Tot.	100.0							
						NNP	70.09	62.15
						NPR	9.83	4.91

[1)] wt. % S in mineral based on the formula $Fe_{0.95}S$ for pyrrhotite

[2)] 0.19 (wt. % S)*31.25/2, as the oxidation of pyrrhotite via oxygen produces only half the amount of H^+ compared to pyrite

[3)] 0.16 (wt. % S)*31.25

[4)] 0.02 (wt. % C)*83.3

[5)] 20 (wt. %)/100*1000 kg/t * 100.09 (g/mol)/216.55 (g/mol) * 0.67

[6)] 6 (wt. %)/100*1000 kg/t * 100.09 (g/mol)/821.16 (g/mol) * 0.67

[7)] 4 (wt. %)/100*1000 kg/t * 100.09 (g/mol)/947.32 (g/mol) * 0.67

[8)] 3 (wt. %)/100*1000 kg/t * 100.09 (g/mol)/300.77 (g/mol) * 0.67

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PREDICTION OF ACID DRAINAGE

- The results suggest that ARD prediction based on SEM mineralogical calculations is at least as accurate as the commonly used static laboratory methods (Karlsson et al. 2018b)
- Compared to this study, in which pulverised rock samples were used and valuable information on the rock texture was mainly lost, the mineralogical prediction accuracy can be further enhanced with a textural investigation and assessment of the intact rocks
  - *For this purpose, the acid rock drainage index (ARDI) has been developed, as part of the geochemistry-mineralogy-texture (GMT) approach (see Parbhakar-Fox et al. 2011).*

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PREDICTION OF ACID DRAINAGE

- New Level 1 methods: scanning drill cores
  - *The toolkit includes application of hyperspectral technologies to derive geoenvironmental domaining index and automated acid rock drainage index values, improved used of handheld tools and chemical tests, data mining, and finding new applications for µCT and 3D XRF drill core scanners.*
- For more information, see:
  - *Cracknell et al. 2018: Automated Acid Rock Drainage Indexing from Drill Core Imagery*
  - *Webinar by Anita Parbhakar-Fox: Waste Characterisation with core scanning technologies, available at https://smi.uq.edu.au/project/gsq-uq-webinar-series*

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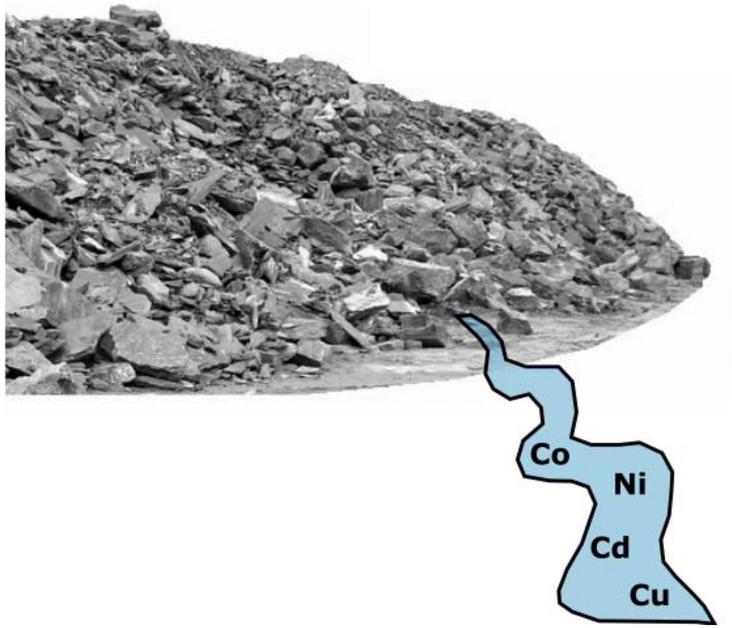
PREDICTION OF METAL MOBILITY

- The mobility of potentially harmful elements from mine waste can be assessed using different selective extraction and leaching methods
- Extraction with aqua regia (AR), a 3:1 mixture of hydrochloric acid and nitric acid, is the most commonly used selective extraction method in mining environment and mineral exploration studies in Finland
- AR is intended to dissolve elements bound especially to sulfide phases, but in addition to sulfides, it also dissolves some silicates, carbonates and secondary precipitate minerals



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PREDICTION OF METAL MOBILITY

- Based on previous studies, the AR-extractable concentrations of waste rocks indicate the elements most likely to be a concern in mine waste drainage (Price et al. 1997; Fosso-Kankeu et al. 2015; Karlsson & Kauppila 2016; Karlsson et al. 2018a)
- In Finland, AR extraction is also the preferred method for evaluating whether mine waste is inert (Finnish Government Decree 2013) and for assessing soil contamination (Finnish Government Decree 2007)

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PREDICTION OF METAL MOBILITY

- A less commonly used method for element mobility prediction is the analysis of leachate from the single-addition NAG test, a test which is primarily designed to determine the potential acid-generation in mine wastes
- As oxidation by hydrogen peroxide (H_2O_2), which is the main reagent in the NAG test, liberates acidity from Fe-sulfides similarly to the sulfide oxidation caused by oxygen, it has been suggested that NAG leachate contents of elements could be useful in assessing contaminant mobility in long-term acid-generating reactions

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PREDICTION OF METAL MOBILITY

- Karlsson et al. 2020 (under review): a study to compare the performance of NAG test leachate analysis and AR extraction in drainage quality assessment, using waste rock and drainage water samples from several Finnish waste rock sites

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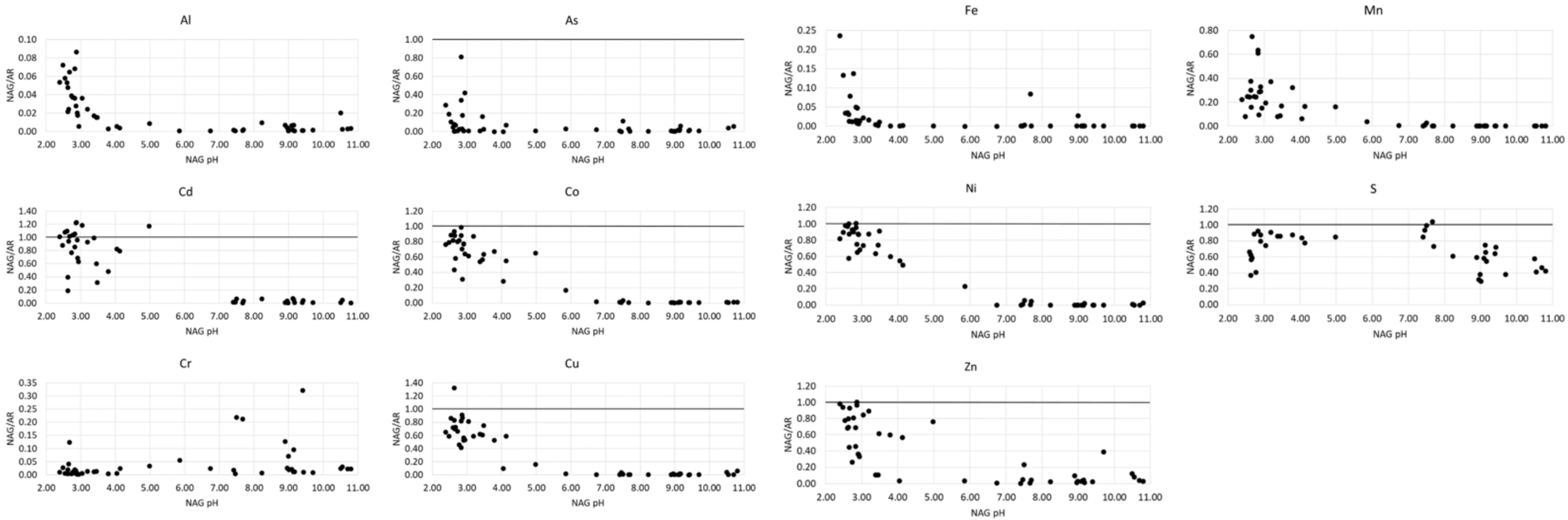
PREDICTION OF METAL MOBILITY

Comparison of AR and NAG test leachate results

- AR more efficient, NAG/AR ratio drops when pH rises



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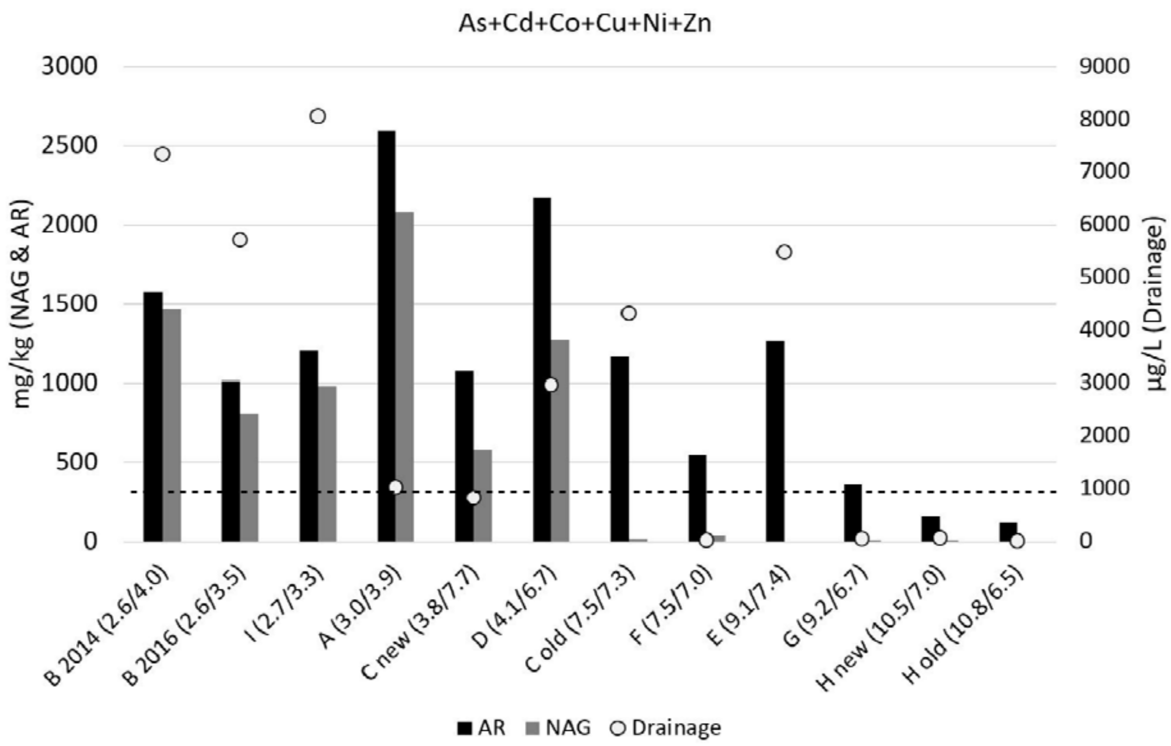
COMPARING AR AND NAG TEST LEACHATE RESULTS TO ACTUAL DRAINAGE CONCENTRATIONS

- Elevated concentrations of harmful elements in the AR-extractable fraction of waste rocks seem to reflect elevated concentrations in drainage water
- If the AR extractable sum of As, Cd, Co, Cu, Ni and Zn is >1 000 mg/kg, there is a high risk for a high-metal (>1 000 µg/L) drainage
- Elevated AR extractable As, Cd, Co, Cu, Ni and Zn concentrations reflected elevated concentrations in drainage water, excluding pessimistic prediction of Al, Cd, Co and Cu in some circum-neutral drainage cases, and Cr in general



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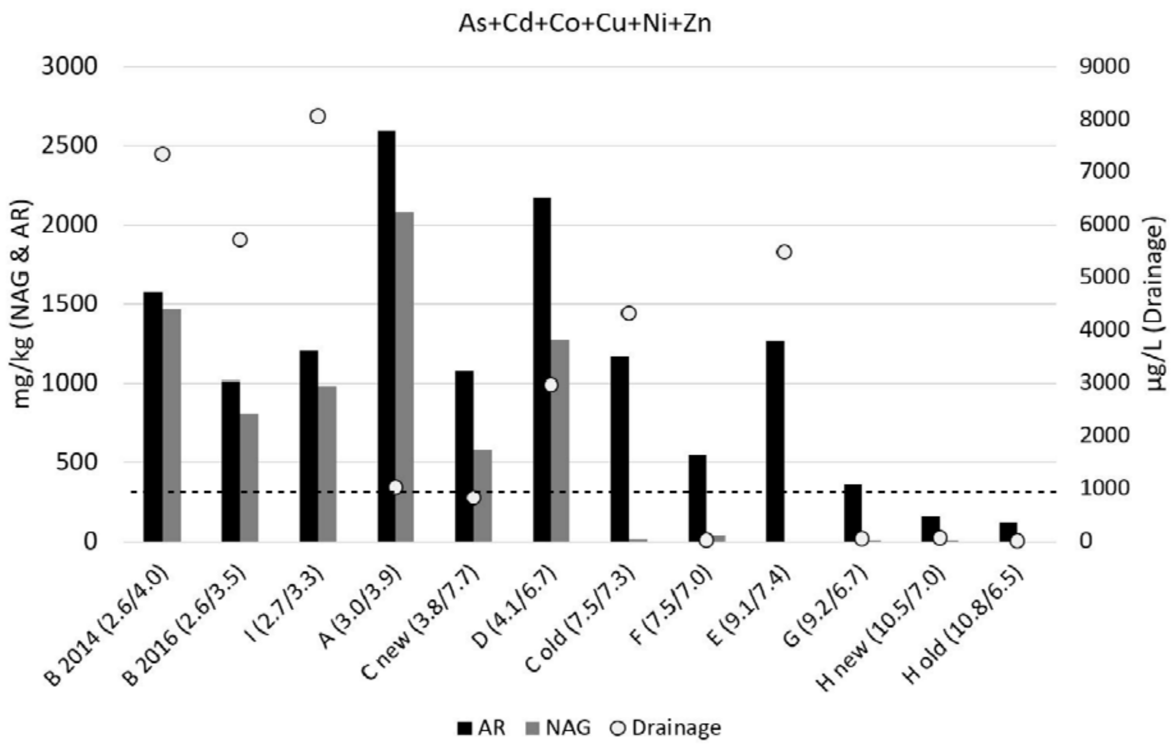
COMPARING AR AND NAG TEST LEACHATE RESULTS TO ACTUAL DRAINAGE CONCENTRATIONS

- Analysis of NAG test leachate was also useful for assessing the mobility of Al, Cd, Co, Cr, Cu, Ni and Zn, but only when the NAG test leachate was acidic
  - *Most elements of interest precipitated when the leachate pH was above 3-6*
- If the sum of As, Cd, Co, Cu, Ni and Zn in NAG test leachate is >500 mg/kg, there seems to be high risk for a high-metal drainage
- On the other hand, <500 mg/kg in NAG test leachate does not exclude the risk for high-metal drainage in circum-neutral drainage systems



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FIELD SCALE KINETIC METHODS

- Reading: Larkins et al. (2016) Design, construction, instrumentation, and monitoring of pilot scale waste rock cover systems: concept review and case studies
  - *Available at: http://tupa.gtk.fi/raportti/arkisto/102_2016.pdf*
- Several lysimeter tests done recently or under way by GTK
  - *Hitura waste rocks, Kevitsa waste rocks, Särkiniemi waste rock cover systems, Rautuvaara biochar cover systems*

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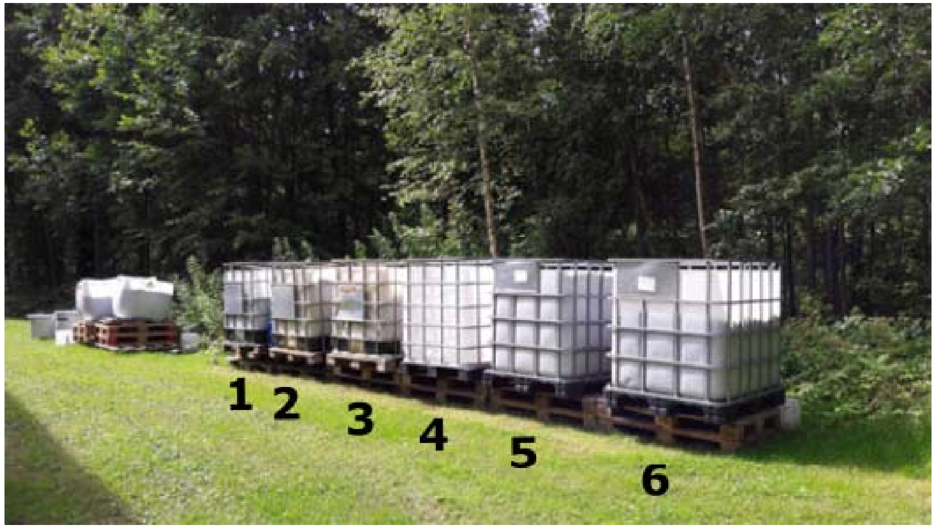
FIELD SCALE KINETIC METHODS

- Hitura waste rocks: using serpentinite to cover acid producing mica shist
- Kevitsa: waste rock utilization potential in construction
- For results, see Karlsson et. Al (2018c) Potential for beneficial reuse of waste rocks from Kevitsa and Hitura mines: indicative data from lysimeter tests
  - *Available at http://tupa.gtk.fi/julkaisu/bulletin/bt_408.pdf*



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FIELD SCALE KINETIC METHODS

- Ongoing test at the Särkiniemi waste rock site:
  - *Utilization of biogas production residue in cover system, comparison to till cover*
  - *Started in 2019*

MODELLING

- Predictive modelling of drainage quality from waste materials combines hydrogeological and geochemical modelling approaches
- Prediction involves a set of different laboratory and field-scale results that are used as a basis and data input for the predictive modelling
- Several different codes for different modelling purposes

Model Type	Codes	References
Empirical/engineering codes	RATAP	MEND 1990
	WATAIL	MEND 1993
	MINTOX	MEND 1997, Bain et al. (2000)
	ACIDROCK	Scharer et al. (1994)
Geochemical codes	WATEQ4F	Ball & Nordstrom (1991)
	MINTEQ/MINTEQA2	Allison et al. (1991)
	MINQL	Schecher and McAvoy (1991)
	PHREEQE/PHREEQM	Parkhurst et al. (1985)/Appelo and Postma (1993)
	GEOCHEM	Parker et al. (1995)
	EQ3/6	Wolery (1992)
	HSC Chemistry	Lamberg & Tommiska (2009)
	CHESS	van der Lee et al. (2003)
Sulphide oxidation/ AMD codes	PYROX	Wunderly et al. (1996)
	FIDHELM	Pantelis 1993
	TOUGH-AMD	Lefebvre et al. (2001)
	TOUGH-CHEM	Xu et al. (2000)
	RETRASO	Saaltink et al. (2002)
	SULFIDOX	Brown et al. (2001)
	THERMOX	da Silva et al. (2009)
	MINTRAN	Walter et al. (1994)
Reactive transport codes	PHREEQC	Parkhurst & Appelo (2013)
	CrunchFlow	Steefel & Lasaga (1994)
	MIN3P	Mayer et al. (2002)
	PHT3D	Prommer et al. (2003)
	PHAST	Parkhurst et al. (2005)
	Geochemist's Workbench	Bethke (1997)
	HYDROGEOCHEM	Yeh and Tripathi (1990)
	TOUGHREACT	Xu and Pruess (2001)
	PHWAT	Mao et al. (2006)
	HYTEC	van der Lee et al. (2003)
	ORCHESTRA	Meeussen (2003)
	eSTOMP	White & Oostrom (2006)
	PFLOTRAN	Lichtner et al. (2015)
	OpenGeoSys(OGS)	Kolditz et al. (2012)
	HP1/HPx	Šimunek et al. (2012)
	RICH-PHREEQC	Wissmeier & Barry (2010)

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MODELLING

- Reading: Muniruzzaman et al. (2018) Water quality prediction of mining waste facilities based on predictive models
  - *Available at: http://tupa.gtk.fi/raportti/arkisto/16_2018.pdf*

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MODELLING

- Despite the individual mechanisms responsible for the generation/propagation of ARD are fairly known, the prediction of overall dynamics in a mine waste system is challenging due to the nonlinearity and multilevel coupling associated with the key physiochemical processes
- Numerical modeling proved to be instrumental in predicting such systems, but there is still a lack of attention towards incorporating the inherent inter- and intra-phase process coupling in those models
- New modelling tool: AMD-PHREEQC by Muniruzzaman et al. (2020, article under review)

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MODELLING: AMD-PHREEQC

- A three-phase multicomponent reactive transport model taking into account the variably saturated water flow, multicomponent advective-dispersive transport both in aqueous and gaseous phase, and chemical reactions including thermodynamic databases
- The model is capable to capture the species-species coupling due to solute/surface charge or gas phase pressure by solving Nernst-Planck equation for the aqueous transport, and Maxwell-Stefan equation for the gaseous transport
- The unsaturated flow and transport code is coupled with PHREEQC, utilizing PhreeqcRM module, to simulate a wide range of reactions included in PHREEQC's package
- The model can be effectively used in environmental risk assessment, long-term drainage quality predictions, and data interpretations in waste dumps

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THANK YOU

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