Multivariate methods for improved geometallurgy sampling

Q. Dehaine[a] and K.H. Esbensen[b]
[a]Geological Survey of Finland (GTK), Circular Economy Solutions Unit, Vuorimiehentie 2, 02151 Espoo, Finland.
E-mail: quentin.dehaine@gtk.fi
[b]KHE Consulting, Copenhagen, Denmark. khe.consult@gmail.com

Geometallurgy is at the core of life-of-mine value chain optimisation, with the aim of integrating geoscientific disciplines along with mining engineering and minerals processing. The objective is to link comprehensive geological, geochemical, mineralogical and geotechnical information with metallurgical and mining variability - based on spatially distributed samples. The spatial coverage is a crucial element in this process. Geometallurgy samples are used for metallurgical testing in the service of plant and process design and optimisation. To avoid discrepancies between the expected and actual process performance, geometallurgical test work must be based on *representative samples* collected and processed in compliance with the Theory of Sampling (TOS). However, even if samples are initially collected to populate a multivariate block model, most of TOS' recommendations for estimating sampling protocols and sample representativeness is *univariate*. While the univariate approach is sufficient when a sample must be representative for one property only *e.g.*, for grade estimation, it fails to properly qualify representativeness of a sample which must be representative for multiple properties such as for geometallurgical purposes. Indeed, a geometallurgy sample is considered representative *sensu stricto* only if its metallurgical behaviour is representative of that of the full zone of the orebody it represents. This can only be achieved if-and-when geo-metallurgical samples are representative for the full set of ore properties that influence process performance. The critical success factor of multivariate representativeness can be assessed using multivariate approaches, such as the multi-variogram, which allow us to summarise the global variability of multiple properties into a single characteristic function. This approach could be optimised by using downstream results from geo-metallurgical process modelling, to select or weight, the individual property contributions to the multi-variogram according to their importance, thereby allowing to optimise a specific geometallurgical sampling procedure in terms of sampling mode, sampling frequency and the number of increments involved according to the overall process performance.

Introduction

The Theory of Sampling (TOS) is inextricably linked to the minerals and mining industry as the source of inspiration for Gy's sampling theory[1]. In the mining, and many other industries, technical and business decisions, as well as project evaluations are heavily dependent on representative sample collection along the entire mine value chain from exploration to closure[2,3]. The optimisation of this value chain over the life-of-mine (LOM) is enabled by the application of geometallurgy[4,5]. Geometallurgy - often reduced to a combination of geology and mineral processing to document the empirical variability within an orebody and to quantify the impact of ore properties onto process performance - is a multi-disciplinary holistic approach aiming at the best possible use of mineral resources in terms of energy and resource efficiency, environmental impact and - of course - revenues, by integrating all relevant geoscientific and engineering disciplines[5,6]. It involves understanding and measuring geological, mineralogical and metallurgical ore properties to generate a database to be integrated into a spatial predictive model for mining operation and mineral processing, as well as mine planning and financial analysis of future or existing mines[7,8]. The aims are to improve resource management, metallurgical processes performance, and ultimately the net present value (NPV) of a mining project, while reducing operational, technical and environmental risks[4,6,9], all aligned with the two UN world goals featured as *lead-motifs* for this WCSB10.

Traditionally, *composite samples* that are reported to be 'representative' of the orebody are collected based on grade and spatial location for metallurgical test work yielding metallurgical parameters (*e.g.*, throughput, recovery) used to design process plant[10]. But in some cases, after the first years of operation, the commissioned process plant can be found not to be performing as planned, which translates into an overrun of the Capital Expenditure (CAPEX) or the money required to build and commission a mining operation run out before the point where it can start producing a saleable concentrate[9]. The most often identified reason is that this approach does not account well enough for spatial variability within the orebody, which translates into unnecessary processing variability over time due to insufficient and unrepresentative material characterisation which has led to inappropriate initial test work. Geometallurgy aims to resolve such unwanted variability, but still requires high-quality metallurgical test work, most emphatically based on representative samples collected and processed within the framework of TOS[11].

Sampling for geometallurgy

Sample Types

Geometallurgy heavily relies on sampling of different sample types, situations, size and scales each with distinct objectives and requirements. In practice four different sampling procedures can be differentiated[12]:

- *In-situ* information which is extracted virtually/digitally from the orebody, for example in the form of geophysical data, downhole measurements such as density, conductivity, or assays.
- *Ex-situ* physical sampling extracted from the orebody, *e.g.*, drill cores, cuttings, or chips.
- *Process sampling* when samples are extracted from a moving stream of ore, regarded as a 1D lot, in the mineral processing

doi: https://doi.org/10.1255/tosf.167

plant. This can be particulate and solid material extracted from a conveyor belt (run-of-mill, gravel, feed, etc.) or pipelines (slurries, process water, etc.).
- *In-line sampling* (in-line sensor measurements) collected from moving streams (belts, pipes, froth product/flotation stations). This includes a wide variety of analytical technologies collectively known as PAT (Process Analytical Technologies) *e.g.*, Raman, IR, XRF, NAA, Camera-based image analysis)[13].

In terms of dimensionality, these sample types can be grouped in two categories: (i) spatially distributed samples (*i.e., in-situ* and *ex-situ* samples) and temporally distributed samples (*i.e.*, process samples and in-line samples). Both categories are critical for the success of the geometallurgy approach, even if spatially distributed samples remain a prerequisite to build the 3Dgeometallurgy model. The importance of each sample type also varies depending on the project status. Spatially distributed samples are critical at the early stage of the project for *strategic geometallurgy* whereas process samples and data are more critical at the operating stage for *tactical geometallurgy*[14]. These two levels of implementation of geometallurgy differ by the required granularity and the decision-making time horizons: strategic geometallurgy focuses on the whole orebody and long-term LOM, whereas tactical geometallurgy has a short- to medium-term operational focus during mining[14].

Sample scale and size

In terms of scale and size, the geometallurgy approach usually employs small spatially distributed samples to perform lab-scale tests which are used as proxies for process parameters which are then compared with larger metallurgical samples to establish correlations prior to modelling[5]. Small process samples are also collected for process control, reconciliation or development[15]. Even larger bulk metallurgical samples, representative of both grade, spatial and population distributions within the ore zone, are collected for pilot testing and plant design[16].

Number of samples

Some generalised recommendations exists for numbers of samples per relevantly identified geometallurgy domains or ore types, but this approach most often require proper analysis on a case-by-case basis, especially as concerns how to sample across intersections[10,17]. Factors like *in-situ* heterogeneity (*e.g.*, grade, alteration, mineralogy and texture), orebody size and number of domains must be taken into considerations and samples evenly distributed[10]. The number of samples required to forecast different geometallurgy parameters varies significantly depending on the targeted parameters (*e.g.*, mass pull, recovery rates, concentrate quality) and the level of reliability required[17]. In practice, the data spacing required is that of an Indicated/Measured resource with the drill grid define via the use of the semi-variogram (referred to as variogram). In most cases however, such variographic analysis is based on univariate grade or elemental assay only[10,18].

Defining a representative geometallurgy sample

A sample can be described as being representative when it results in acceptable levels of bias and precision[19]. Hence, besides sample type, size and number, one of the main difference between sampling for geometallurgy compared to traditional sampling program in the minerals and mining industry, is that samples must be representative for several properties as opposed to one parameter only as is tradition (*i.e.*, the grade)[20]. Indeed, by definition geometallurgy is multivariate (multi-component) as it aims at predicting and quantifying the full multi-component metallurgical performance based on geological, geochemical, textural and mineralogical ore characteristics, which is hardly optimal when considering only one analyte[21]. Indeed geo-metallurgical samples are always collected with the purpose of acquiring multivariate data through a comprehensive set of measurements (*e.g.*, chemical or mineralogical assay, hardness) or testing (*e.g.*, Bond Work index, kinetic flotation test, leaching test)[10,18]. The resulting multivariate dataset is then either integrated into a 3D block model, when dealing with drill core samples for instance, or with a process model, when dealing with process sampling. In either case, the outcome of the model, such as the mining bloc model value or the simulated process performance, is directly dependant on the quality of the input variables of which there are always *many*. Thus, the issue of multivariate representativeness is at the core of geo-metallurgical sampling. This is especially relevant concerning optimal definition of operative Decision Units (DU), see below. It’s fair to say that this issue is a work in progress, very challenging and therefore very interesting.

Spatial coverage – inferential statistics to the rescue

The overarching problem in geometallurgy is how to design a sampling campaign that guarantees the necessary-and-sufficient *spatial coverage* of the entire mineralisation or orebody? Many of the elements involved in a general solution are known to the geometallurgical realm, but the critical success factor will always be **how to** sample a 3-D body, or a 1-D drill core (if you can sample a 1-D core adequately, you can sample any number, going a long way towards a full 3-D body). Though always strongly dependent upon the specific orebody, the general problem is that one cannot sample the entire (1-D, 3-D) body, however desirable *would be* full sampling, full coverage, full certainty, full confidence in the ensuing test work. One always must *sample* in time (dynamic lots) and/or space (stationary lots), *e.g.*, what fraction of the possible total number of samples that *could* be extracted in a lot, are needed to make a satisfactory geometallurgical characterisation with respect to a desired confidence and reliability? For example, it is up to project management to decide *a priori* its desired confidence level (X %) that no more than Y % of samples *may* fail a specific quality criterium, *e.g.*, may exceed an analyte or component Z maximum concentration threshold.

Bringing in a modicum of statistical rigor, prior to any sampling event, an operative Decision Unit (DU) must be established; the DU is the material volume that an analytical result makes inference to. A lot is a collection (population) of individual DUs that will be treated as a whole (accepted or rejected), depending on the analytical results for individual Decision Units. The application of the Theory of Sampling (TOS) is critical for sampling the material within a Decision Unit. However, knowledge of the analytical concentration of interest *within* a Decision Unit will not necessarily provide information on *unsampled* Decision Units, especially for heterogenous (or very heterogeneous) lots like many mineralisations and orebodies, where DUs can be of very dissimilar characteristics. The very geometallurgy variability issue spills over into the critical issue of case-relevant DU definition. While this issue undoubtedly looms large in many geo-metallurgical projects, there are no universally applicable rules at this time.

In such geo-metallurgical cases where every DU cannot possibly be sampled but where the spatial coverage demand is always marked, application of *non-parametric statistics* can be used to make inference *from* sampled Decision Units *to* Decision Units that are *not* sampled. The combination of the TOS for sampling of individual Decision Units along with non-parametric statistics offers the best possible inference for situations where there are more Decision Units than can practically be sampled physically. Recently Ramsey and Esbensen (2022) presented this combined TOS-statistics sampling scope in a fully worked-out framework, ready to be taken into the sampling realm, including geometallurgy[22]; the title tells it all: "Inferential statistical sampling of hyper-heterogeneous lots with hidden structure: the importance of proper Decision Unit definition".

Theory of Sampling (TOS) and variographic analysis

TOS and univariate variographic analysis

Introducing TOS and variographic analysis in the context of this paper for the World Conference of Sampling and Blending, WCSB10) must surely be one of the most unnecessary tasks conceivable. Suffice to direct attention to no less than three recent textbooks[23–25], all three conveniently presented in a comparison format TOS Forum, issue 10 (2020)[26]. Variographic data modelling has been presented in very many contexts (not least in the three textbooks referred to above), but also specifically for the present audience in Minnitt & Esbensen 2018[27] and in Pitard and Minnitt 2008[28]. The reader will find a plethora of further references and scores of application examples in these five exposés.

For the present scope suffice to point to the imperative of representative sampling procedures overall, whether directed at stationary or moving (process) lots. On this basis, a variographic data model allows powerful insight into how well a particular sampling system/procedure/solution has succeeded in eliminating and/or reducing maximally all detrimental Incorrect Sampling Errors (ISE), Correct Sampling Errors (CSE) as well as the Total Analytical Error (TAE) effects on the analytical results. Variographic data modelling is enormously powerful and comprehensive – indeed it has only one negative to it, it is manifestly only univariate. Only!

TOS and multivariate variographic analysis

Although, the need for TOS to enter the multivariate realm was already exposed at WCSB2[29], application of TOS so far have been almost exclusively been univariate[11]. However perfunctory multivariate approaches are well-known in two disciplines related to geology, geochemistry, mining, mineral processing *a.o.*, *geostatistics* and *chemometrics*, and it is from these disciplines that two solutions have recently been proposed to integrate the multivariate nature of heterogeneity with TOS.

An initial solution derived from the world of chemometrics is to reduce the dimensionality of the dataset through application of Principal Component Analysis (PCA) and to model a variogram on the *scores* of the first few principal components[30]. This approach, referred to as *variogram (t-score)*, offer the advantage of combining a variable reduction procedure that describes the correlation between all the variables involved in the multivariate data and which highlights the hidden structures and spatial (or temporal) patterns through variables grouping (the PCA) to a procedure that characterises autocorrelations within an ordered dataset, the variogram[31]. Alternatively, a 'reverse' approach, referred to as *PCA (variograms)*, consists in applying PCA analysis on individual variograms for each variable, which allows to conduct similar data analytical interpretations and results, but with the benefits of knowing the individual variability characteristics of each individual variables[32,33]. Application of this complementary 'dual' approach has to be guided by the specifics of a particular context[22-25].

Another approach, derived from the world of geostatistics, which recently was introduced to the realm of TOS (WCSB7) is the multivariate variogram, also referred to *multivariogram*[34]. In multivariate variographics, each measured variable x_i (*e.g.*, chemical analytes, physical properties) is considered collectively as one multivariate dataset and combined in one vector, X, with p elements (the p individual variables). The multivariogram V_j of X is then calculated using the master equation[20]:

$$V_j(X) = \frac{1}{2(N-j)} \sum_{i}^{N-j} \left(X_i - X_{i+j}\right) M \left(X_i - X_{i+j}\right)^t, \qquad j = 1, \cdots, N/2 \tag{1}$$

(subscript t is the transpose operator), N is the total number of increments (*i.e.*, samples) collected, j the process lag parameter, and M a metric (positive definite p x p matrix) defined as the inverse of the variance-covariance matrix of X. M corresponds to the Mahalanobis distance (MD), which defines the "distances" between the units, *i.e.*, the relationship between the variables, takes into account the correlation in the data[35,36] and is considered to be adapted to multivariate variography as opposed to the Euclidean metric for which the multivariogram would only be the *sum* of the univariate variogram of each individual variable[20,34]. The multivariogram can thus be used to summarize the overall spatial (or temporal) variability of data from a set of variables in one structural function and thus highlight the spatial (or temporal) structures that are *common* to these variables[20].

The added value of multivariate variographic approaches for geometallurgy sampling

Alas, there has been limited amounts of publication on multivariate variographic analysis for geometallurgy sampling, most of them focusing on process sampling. One of the first study on the topic, applied to industrial residue stream considered for by-product metal recovery[37], applied and combined the above-mentioned multivariate approaches on a set of 8 variables (Figure 1). These variables reflects critical properties of the residue with regards to the design of a by-product recovery process[38,39] and are also used in in the establishment of a geometallurgy model[40]. The use of the multivariogram allowed to assess the true global variance of the sampling error and thus design the optimal sampling protocol with respect to all the variables of interest. However, the estimated global variance with this approach is very high. This mean that designing a sampling campaign, and defining the number of increments to be samples, based on the multivariogram would lead to (unrealistically) large number of increments to be sampled to obtain a reasonable sampling variation. To overcome this issue the authors proposed to combine the multivariogram with PCA analysis by computing the multivariogram of the first PCs' scores. This allows to reduce the influence of noisy data and thus reduces the overall sampling variance.

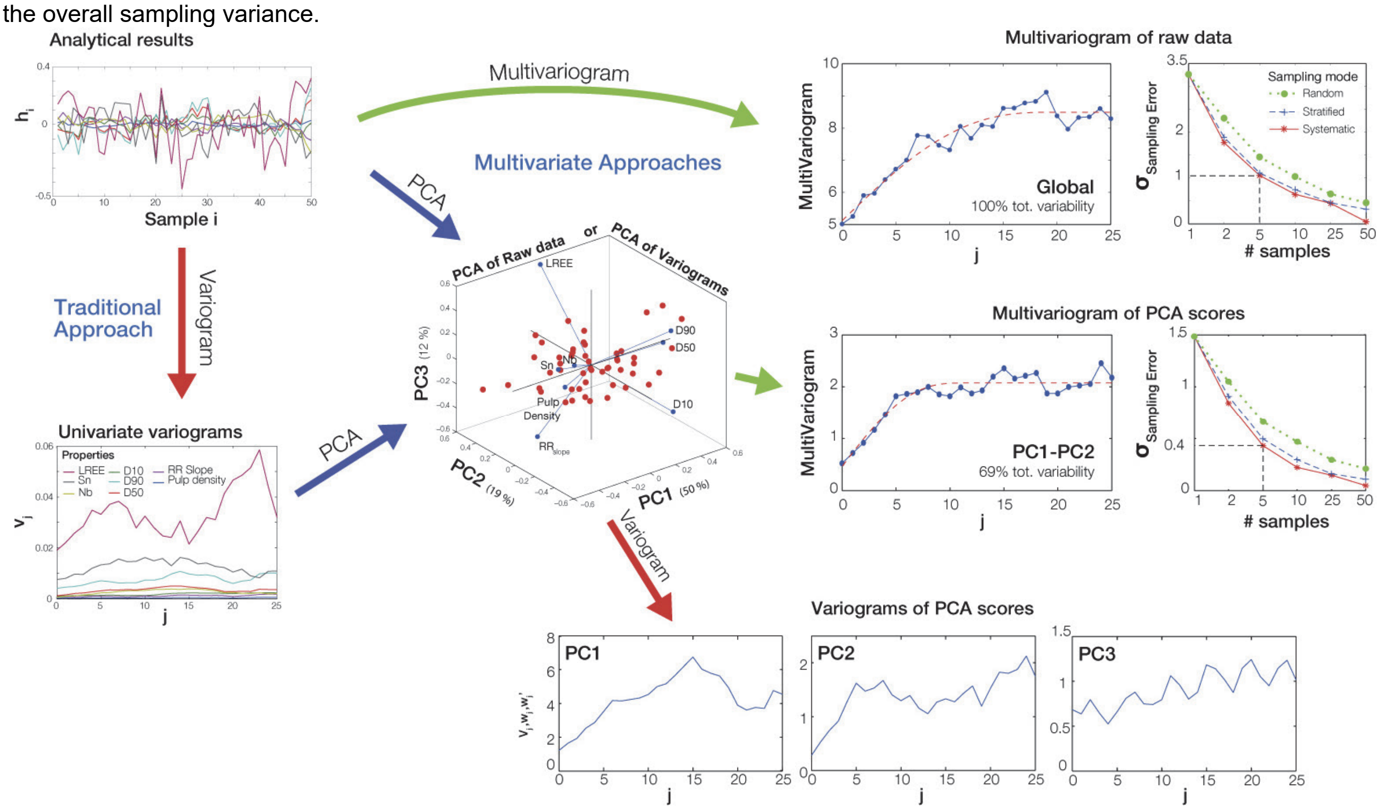

Figure 1. Graphical abstract illustrating the univariate *vs* multivariate variographic approaches for process sampling explored in Dehaine *et al.* (2016)[20].

A similar approach was recently tested in water management for process water quality monitoring and environmental purposes[41]. Water quality is indicated by a large number of physico-chemical properties that must be monitored through time. The use of multivariate statistics allow to reducing the number of monitor charts needed, increase the signal to noise ratio while taking into consideration all properties and their correlation[42]. Therefore, the authors tested the use of multivariate approach to design sampling procedure for water quality control. The multivariographic analysis revealed the hidden cyclic variation through its ability to summarize the time variations and the correlation between multiple variables that were not visible through the classical univariate variogram approach. Similarly, to the previous case study[20], the number of increments recommended by the global multivariogram is impractically high, but can be reduced by combining PCA to the mutivariogram. This study highlights the benefits of using multivariate variography to improve water sampling procedures in the mining industry and to reduce both operational and environmental risks associated with water quality variability.

However, despite the reduction in global sampling variance through the use of PCA and even when choosing the properties of interest carefully, the resulting global variance obtained with the multivariogram may remain very high. In particular, some variables that contribute to a major proportion of the global (multivariate) variability could be less important for the process performance than others having a lower variability. Indeed, when sampling for geometallurgy testing, the sample can be considered as being representative when it results in acceptable levels of bias and precision *for the outcome of test* meaning the metallurgical performance index.

To address this issue, a new approach has been proposed at WCSB8, combining the multivariogram with process modelling and multivariate data analysis methods such as partial least squares (PLS) regression[43]. The approach was tested on industrial kaolin plant using sensor data as key process variables and a predictive process performance model based on PLS regression (Figure 2). The study showed that the PLS model regression coefficients can be used to weigh the variables according to their relevance for the process in a weighted metric to design an optimised sampling procedure in terms of frequency, sampling mode and number of increments according to the actual overall process performance. This approach has potentially many applications in geometallurgy as it would allow to tailor the metric used in the multivariogram according to the objective of the metallurgical test using existing physical or experimental models (Multiple Linear Regression, PLS, Design of Experiments). This would therefore allow to increase the representativeness of geometallurgy samples and decrease the risks associated with metallurgical performance variability.

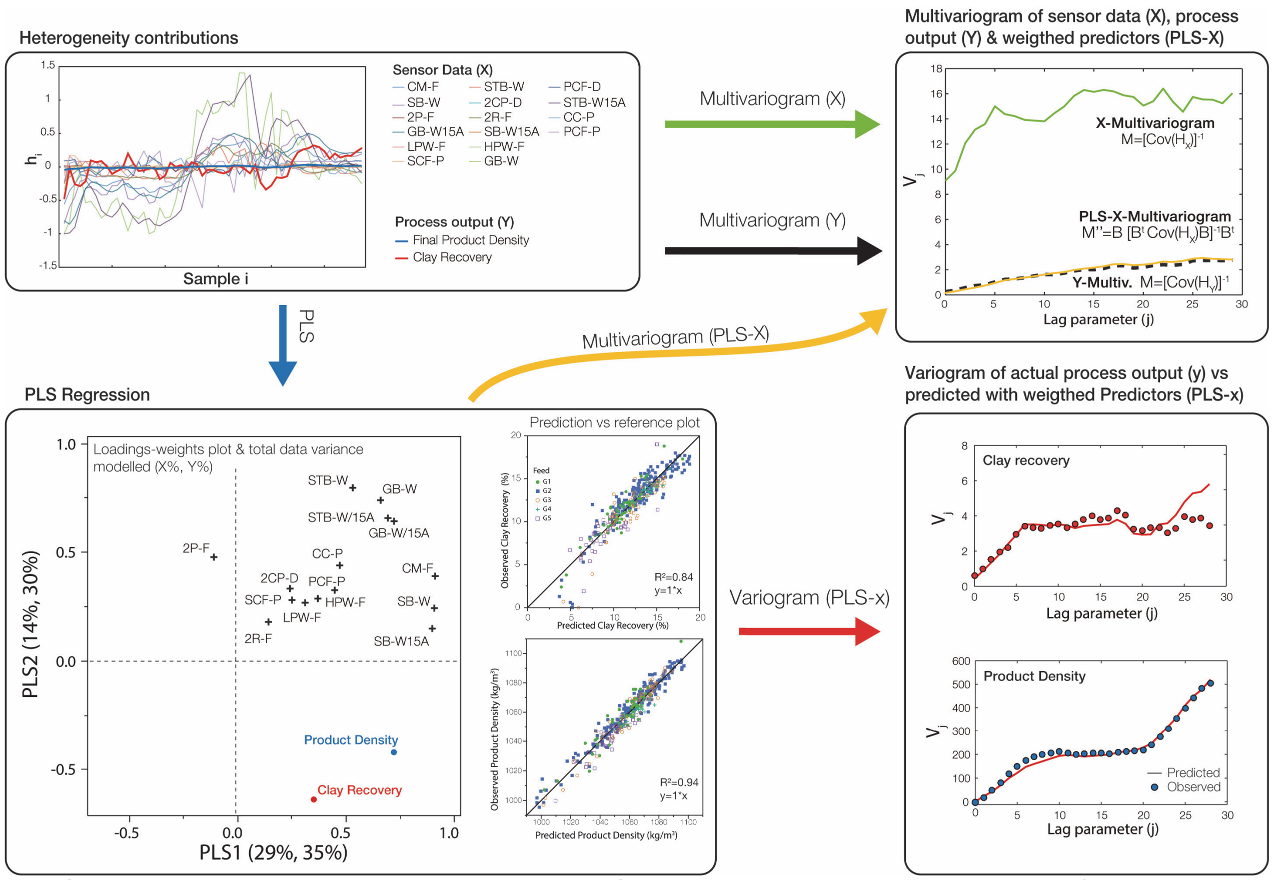

Figure 2. Graphical abstract illustrating the combined PLS-multivariate variographic approaches for process sampling explored in Dehaine *et al.* (2017)[43].

Discussion and conclusion

The examples described above show the strong benefits stemming from acknowledging that material heterogeneity as well as process heterogeneity is inherently multivariate in nature.

Acknowledging the multivariate nature of mineralisation and orebody heterogeneity, and the complex interactions involved in plant performance as well as the mining operation influence over the entire LOM, is at the essence of geometallurgy. It is therefore essential to reap the benefits from starting to integrate the multivariate scope during the design of geometallurgy sampling campaigns and not only for process sampling. But there is a long way to go, and much to be learned by all parties. It is certainly not just a matter of applying multivariate data analysis to any multi-variable, or multi-parameter assemblage encountered; there is an ongoing need for geological/mineralogical/geochemistry knowledge to support the multivariate scope. It is likely that the basic univariate scope will always be needed for fully comprehensive geometallurgy.

So, while only in its first stages within many geoscience fields, we predict an increasing role for the multivariate approach to almost all technological fields and application areas in which sampling plays a role. There is powerful insight to be gained by properly applied univariate variographic in technology and industry, *e.g.,* see the many examples in Engström, K.: "Sampling in Iron Ore Operations – Evaluation and Optimisation of Sampling Systems To Reduce Total Measurement Variability"[44] - and there is disruptive power in transgressing to a true multivariate scope[41,43].

Call to action

The multivariate approach has been described in adequate depth above – to present our main errand: This WCSB10 presentation is an ***invitation*** to all parties who would take an interest in venturing *outside* the conventional univariate box. An invitation to collaboration for pushing the envelope of sampling also into the multivariate realm … You are invited – you are welcome!

ORCID iDs

A. Q. Dehaine: https://orcid.org/0000-0003-4674-3187
B. K.H. Esbensen: https://orcid.org/0000-1111-2222-3334 Full publication list here: https://kheconsult.com/

References

1. P. Gy, "Part V: Annotated literature compilation of Pierre Gy", *Chemometrics and Intelligent Laboratory Systems*. **74**, 61-70 (2004). https://doi.org/10.1016/j.chemolab.2004.05.010
2. K.H. Esbensen, "TOS reflections: is there a third way? (to promote the Theory of Sampling)", *TOS Forum*, 21-23. https://doi.org/10.1255/tosf.122
3. Q. Dehaine, "Loosen the TOS stipulations and face the economic consequences", *Spectroscopy Europe*. **33**, 32-33 (2021).

https://doi.org/10.1255/sew.2021.a37
4. S.C. Dominy, L. O'connor, A. Parbhakar-Fox, H.J. Glass, and S. Purevgerel. "Geometallurgy—A route to more resilient mine operations". *Minerals* **8**, 560 (2018). https://doi.org/10.3390/min8120560
5. S.C. Dominy and L. O'Connor, "Geometallurgy – Beyond Conception", in *Proceedings of the 3rd AusIMM International Geometallurgy Conference*. AusIMM, pp. 3-10 (2016).
6. R. Pell R, L. Tijsseling, K. Goodenough, F. Wall, Q. Dehaine, A. Grant, D. Deak, X. Yan and P. Whattoff, "Towards sustainable extraction of technology materials through integrated approaches". *Nature Reviews Earth & Environment.* **2**, 665–679 (2021) https://doi.org/10.1038/s43017-021-00211-6
7. C. Lund and P. Lamberg, "Geometallurgy – A tool for better resource efficiency". *European Geologist*. **37**, 39-43 (2014). https://doi.org/10.1039/C4SM02815E
8. S.R. Williams and J.M. Richardson, "Geometallurgy mapping: a new approach that reduces technical risks", in *Proceedings of 36th Annual Meeting of the Canadian Mineral Processors Conference*. CIM, pp. 241-268 (2004).
9. S. P. Michaux and L. O'Connor, "How to Set Up and Develop a Geometallurgy Program", *GTK Open Work File Report 72/2019* (2020).
10. S.C. Dominy, L. O'Connor and Y. Xie, "Sampling and Testwork Protocol Development for Geometallurgy Characterisation of a Sheeted Vein Gold Deposit", in *Proceedings of the 3rd AusIMM International Geometallurgy Conference*. AusIMM, pp. 97-112 (2016).
11. K.H. Esbensen, "Theory of Sampling - what's next?", in: *Proceedings 8th World Conference on Sampling and Blending (WCSB8)*. AusIMM, pp. 29-38 (2017).
12. R. Baumgartner, "Geometallurgy – Optimising the resource", in *Ore Dressing, Geometallurgy and Environmental Geochemistry of Mine Waste*. pp. 1-41 (2012).
13. K. A. Bakeev, "Process Analytical Technology: Spectroscopic Tools and Implementation Strategies for the Chemical and Pharmaceutical Industries". 2nd ed, John Wiley & Sons (2010).
14. N. McKay, J. Vann, W. Ware, W. Morley and P. Hodkiewicz, "Strategic and Tactical Geometallurgy - a systematic process to add and sustain resource value", in *Proceedings of the 3rd AusIMM International Geometallurgy Conference*. AusIMM, pp. 29-36 (2016).
15. R.J. Holmes. "Correct sampling and measurement—the foundation of accurate metallurgical accounting", *Chemometrics and Intelligent Laboratory Systems.* **74**, 71-83 (2004). https://doi.org/10.1016/j.chemolab.2004.03.019
16. S.C. Dominy, L. O'Connor, H.J. Glass, S. Purevgerel and Y. Xie, "Towards representative metallurgical sampling and gold recovery testwork programmes", *Minerals*. **8**, 193 (2018). https://doi.org/10.3390/min8050193
17. V. Lishchuk, P. Lamberg and C. Lund, "Evaluation of sampling in geometalurgical programs through synthetic deposit model" in *Proceedings of the XXVIth International Mineral Processing Congress (IMPC)*. pp. 1-11 (2016).
18. S.C. Dominy, L. O'Connor, Y. Xie and H.J. Glass, "Geometallurgy sampling protocol validation by bulk sampling in a sheeted vein gold deposit", in *Proceedings of the 8th World Conference on Sampling and Blending (WCSB8)*. AusIMM, pp. 195-196, (2017).
19. F.F. Pitard, "Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sampling Correctness, and Statistical Process Control". 2nd ed., CRC Press (1993).
20. Q. Dehaine, L.O. Filippov and J.J. Royer, "Comparing univariate and multivariate approaches for process variograms: A case study", *Chemometrics and Intelligent Laboratory Systems*. **152**, 107-117 (2016). https://doi.org/10.1016/j.chemolab.2016.01.016
21. Q. Dehaine, L.T. Tijsseling, H.J. Glass, T. Törmänen and A.R. Butcher, "Geometallurgy of cobalt ores: A review". *Minerals Engineering*. **160**, 106656 (2021). https://doi.org/10.1016/j.mineng.2020.106656
22. C. Ramsey and K.H. Esbensen, "Inferential statistical sampling of hyper-heterogeneous lots with hidden structure: the importance of proper Decision Unit definition", *Spectroscopy Europe*. **34**, 25-32 (2022). https://doi.org/10.1255/sew.2022.ax
23. K.H. Esbensen, "Introduction to the Theory and Practice of Sampling". IM Publications Open (2020). https://doi.org/10.1255/978-1-906715-29-8
24. F. Pitard, "Theory of Sampling and Sampling Practice" 3rd ed. CRC Press, (2019).
25. G. Lyman, "Theory and Practice of Particulate Sampling: An Engineering Approach". Materials Sampling & Consulting (2019).
26. K.H. Esbensen KH. TOS Forum Issue 10, (2020).
27. R.C.A. Minnitt and K.H. Esbensen, "Pierre Gy's development of the Theory of Sampling: a retrospective summary with a didactic tutorial on quantitative sampling of one-dimensional lots", *TOS Forum*. 7-19 (2017). https://doi.org/10.1255/tosf.96
28. R.C.A. Minnitt and F.F. Pitard, "Application of variography to the control of species in material process streams: %Fe in an iron ore product", *Journal of the Southern African Institute of Mining and Metallurgy*. **108**, 109-122 (2008).
29. K.H. Esbensen, "A multivariate perspective on Gy's Theory of Sampling – first foray", in *Proceedings 2nd World Conference on Sampling and Blending (WCSB2)*. AusIMM, p. 9 (2005).
30. M.A. Oliver and R.A. Webster, "Geostatistical basis for spatial weighting in multivariate classification", *Mathematical Geology* **21**, 15-35 (1989). https://doi.org/10.1007/BF00897238
31. P.O. Minkkinen and K.H. Esbensen KH, "Multivariate variographic versus bilinear data modelling" *Journal of Chemometrics*. **28**, 395-410 (2014). https://doi.org/10.1002/cem.2514
32. Z. Kardanpour, O.S. Jacobsen and K.H. Esbensen, "Local versus field scale soil heterogeneity characterization – a challenge for representative sampling in pollution studies", *SOIL Discussions*. **2**, 619-645 (2015). https://doi.org/10.5194/soild-2-619-2015

33. Z. Kardanpour, O.S. Jacobsen and K.H. Esbensen, "Soil heterogeneity characterization using PCA (Xvariogram) - Multivariate analysis of spatial signatures for optimal sampling purposes", *Chemometrics and Intelligent Laboratory Systems*. **136**, 24-35. (2014) https://doi.org/10.1016/j.chemolab.2014.04.020
34. Q. Dehaine and L.O. Filippov, "A multivariate approach for process variograms", in *Proceedings 2nd World Conference on Sampling and Blending (WCSB2), TOS Forum*, 169-174 (2014). https://doi.org/10.1255/tosf.76
35. G. Bourgault and D. Marcotte, "Multivariable variogram and its application to the linear model of coregionalization", Mathematical *Geology*. **23**, 899-928 (1991). https://doi.org/10.1007/BF02066732
36. R. De Maesschalck, D. Jouan-Rimbaud and D.L. Massart. "The Mahalanobis distance". *Chemometrics and Intelligent Laboratory Systems*. **50**, 1-18 (2000). https://doi.org/10.1016/S0169-7439(99)00047-7
37. Q. Dehaine and L.O. Filippov, "Rare earth (La, Ce, Nd) and rare metals (Sn, Nb, W) as by-product of kaolin production, Cornwall: Part1: Selection and characterisation of the valuable stream", *Minerals Engineering*. **76**, 141-153 (2015). https://doi.org/10.1016/j.mineng.2014.10.006
38. Q. Dehaine, L.O. Filippov and R. Joussemet, "Rare earths (La, Ce, Nd) and rare metals (Sn, Nb, W) as by-products of kaolin production – Part 2: Gravity processing of micaceous residues", *Minerals Engineering*. **100**, 200-210 (2017). https://doi.org/10.1016/j.mineng.2016.10.018
39. L.O. Filippov, Q. Dehaine and I.V. Filippova, "Rare earths (La, Ce, Nd) and rare metals (Sn, Nb, W) as by-products of kaolin production – Part 3: Processing of fines using gravity and flotation", *Minerals Engineering*. **95**, 96-106 (2016). https://doi.org/10.1016/j.mineng.2016.06.004
40. Q. Dehaine, L.O. Filippov, H.J. Glass and G.K. Rollinson, "Rare-metal granites as a potential source of critical metals: A geometallurgy case study", *Ore Geology Reviews*. **104**, 384-402 (2019). https://doi.org/10.1016/j.oregeorev.2018.11.012
41. T.M.K. Le, Q. Dehaine, B. Musuku, N. Schreithofer and O. Dahl, "Sustainable water management in mineral processing by using multivariate variography to improve sampling procedures", *Minerals Engineering*. **172**, 107136 (2021). https://doi.org/10.1016/j.mineng.2021.107136
42. T.M.K. Le, M. Mäkelä, N. Schreithofer, O. Dahl, "A multivariate approach for evaluation and monitoring of water quality in mining and minerals processing industry", *Minerals Engineering*. **157** (2020). https://doi.org/10.1016/j.mineng.2020.106582
43. Q. Dehaine, L.O. Filippov and H.J. Glass, "Optimising multivariate variographic analysis with information from multivariate process data modelling (Partial Least Squares Regression)", in: *Proceedings of the 8th World Conference on Sampling and Blending (WCSB8)*. AusIMM, pp. 381-389 (2017).
44. K. Engström, "Sampling in iron ore operations: Evaluation and optimisation of sampling systems to reduce total measurement variability", PhD Thesis, Aalborg University. 2018. https://doi.org/10.5278/vbn.phd.eng.00068