A NEW TOOL FOR THE EVALUATION OF ENVIRONMENTAL SAMPLES: DEVELOPMENT OF SOFTWARE FOR MORPHOLOGICAL AND ELEMENTAL DATA EVALUATION

K. Vilece, M. Schoeppner, A. Larson, M. Nikkinen, C. Norman, M. Lafitte, J. Baute
International Atomic Energy Agency
Wagramerstrasse 5, Vienna A-1400 Austria
E-mail: K.Vilece@IAEA.org, M.Schoeppner@IAEA.org, A.Larson@IAEA.org,
M.Nikkinen@IAEA.org, C.Norman@IAEA.org, M.Lafitte@IAEA.org, J. Baute@IAEA.org

ABSTRACT

The collection of environmental swipe samples by nuclear safeguards inspectors of the International Atomic Energy Agency (IAEA), sample analysis by the IAEA Network of Analytical Laboratories (NWAL), and subsequent evaluation by IAEA evaluators are vital to providing assurances regarding the absence of undeclared nuclear materials and/or activities. The evaluation of such environmental swipe samples includes the assessment of key signatures measured by a variety of instruments and techniques. Particle and bulk isotopic analysis results via mass spectrometry are most commonly used but for many samples morphological and elemental data via the scanning electron microscope (SEM) is also available and useful. This supplementary data can help provide important information about the materials and activities at locations sampled and includes elemental composition, compound identification, as well as morphological images indicating the form, surface characteristics, size, etc., of particulate. The IAEA has designed a simple but effective particle library format and populated the library with images and data for various types of nuclear material (NM) and relevant materials from the nuclear fuel cycle (NFC). A new software tool has been created which is compatible with the new library and allows for effective comparison, search, and more effective evaluation of historical and new particle signatures. The new particle library and software tool will help IAEA evaluators make better use of SEM data in the future.

Keywords: environmental swipe samples, SEM, morphological and elemental data, elemental composition, compound identification, morphological images, particle library, particle signatures

1. INTRODUCTION

International Atomic Energy Agency (IAEA) safeguards includes verification of State declarations regarding nuclear material accountancy via independent analysis of safeguards nuclear material (NM) samples collected by the IAEA. Analysis focuses on uranium (U) and plutonium (Pu) concentrations and isotopic abundances. Analytical results are evaluated statistically to assess the correctness of declarations regarding nuclear material inventory and flow. In the 1990s the discovery of a clandestine nuclear weapons program in Iraq indicated a need for new measures to detect undeclared activities. During the process of strengthening and streamlining IAEA safeguards, the need for analytical techniques to detect indications of possible undeclared nuclear activities or undeclared NM was identified [1].

The usefulness of environmental sampling (ES) was confirmed during a series of field trials in the development programme to strengthen safeguards, entitled “Programme 93+2”. ES swipes were first taken in States with comprehensive safeguards agreements in 1996 and during complementary access (CA) under additional protocols in 1998. Since then, samples have been taken in a large number of States including special sampling campaigns in various States [2].

In 1996 the IAEA SAL Clean Laboratory (SAL/CL) was established and since then numerous laboratories have joined the IAEA Network of Analytical Laboratories (NWAL). Currently, particle and bulk analysis of ES swipe samples is carried out at the IAEA’s Environmental Sample Laboratory (ESL) and NWAL laboratories in the USA, EU (France and Germany), Japan, Russian Federation, UK, Australia, Brazil, South Korea, and China. Over the course of more than twenty-five years, environmental sampling techniques have been further developed by the IAEA with support from Member States.

Today swipe samples are regularly collected during routine inspections, design information verification, and CA. The presence of trace levels of actinides such as U and Pu and their isotopic composition (determined primarily by mass spectrometry) may indicate past or existing nuclear activities. Other analytical techniques are used which focus on specific elements, compounds, and morphologies which may better characterize NM or materials related to the nuclear fuel cycle (NFC). This paper reviews recent efforts at the IAEA to create a particle library encompassing this elemental and morphological data, software to bring this library to life, and to recommend future efforts to allow for automated analysis of morphological images.

1.1. Analytical Techniques

As the IAEA's analytical needs have grown, analytical capabilities of the NWAL have adapted to provide the necessary support. Some areas of support which are performed on a regular basis include:

- High Resolution Gamma Spectrometry (HRGS) as a screening technique for the presence of gamma-emitting radioisotopes;
- X-ray Fluorescence (XRF) as screening technique for the presence of U;
- Bulk analysis for the determination of U and Pu masses and U and Pu isotopics;
- Fission Track Thermal Ionization Mass Spectrometry (FT-TIMS) for the characterization of U and Pu isotopics;
- Secondary Ion Mass Spectrometry (SIMS) particle analysis for the characterization of U isotopics;
- Large Geometry SIMS (LG-SIMS). This technique solves several key measurement problems inherent in conventional SIMS instruments, allows for the measurement of large numbers of particles, and provides U minor isotope performance which is comparable to the FT-TIMS technique.

In addition, other techniques are requested only for specific samples, often involving more dynamic analysis by the laboratories:

- Bulk analysis for the measurement of Am, in particular the amount of ^{241}Am;
- Age-dating of U particles via the measurement of the ^{230}Th/^{234}U isotope ratio;
- Special nuclear forensic analysis. These requests often require the determination of elemental composition (including the level of purity), identification of certain compounds, and characterization of morphological signatures. This analysis is primarily done via SEM with various detector and software systems.

1.2. Increasing Need for Special Nuclear Forensic Analysis

Samples taken during routine inspections and DIV visits are most often from established nuclear facilities such as enrichment plants and facilities with hot cells; isotopic analysis of U and/or Pu-containing particles are therefore most commonly requested. However, increasing numbers of samples are taken during CAs at a more diverse set of installations, including established nuclear facilities, but also at universities, research centers, and many locations which can have a broad range of operations and may have little or no inventory of NMs. This results in more diverse samples and analytical tasks which often require a larger set of sample processing methods, analytical techniques, evaluation tools, and evaluation challenges.

The number of ES swipe samples taken annually since 1996 is shown in Figure 1. The average sample load per year is seen to be about 400 with the 2020 number closer to 500. About 40% of all swipe samples have been taken during CA during the last five years. Over the last 25 years, significant numbers of ES samples have been analysed by elemental and morphological techniques producing a large amount of data from a wide variety of NM and NFC materials. This data is associated with a large variety of facilities and/or sampling locations. With an increasing number of samples requiring a more "investigative" approach, elemental and morphological data is becoming more common and useful for the evaluation of samples collected by the IAEA.

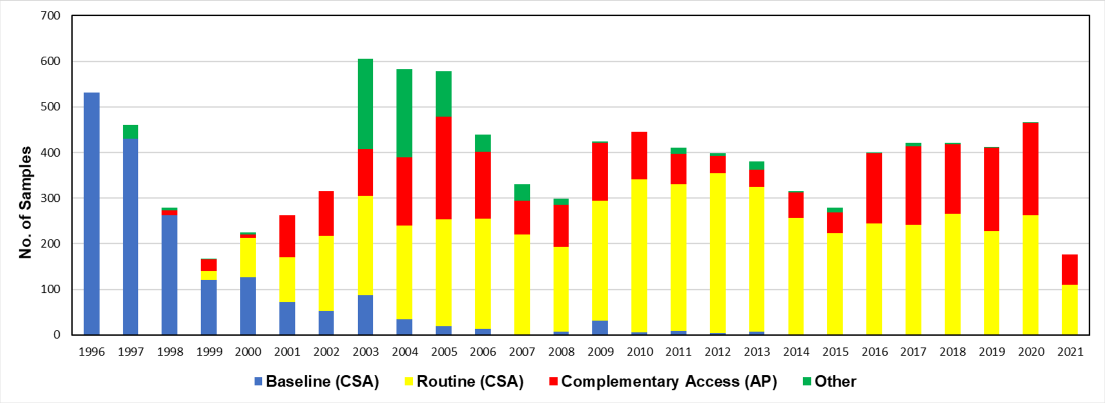


Figure 1: ES swipe samples taken from 1996-2021 (as of July 2021) categorized as baseline, routine, CA, or other. A significant portion of ES is now taken during CA visits.

2. ELEMENTAL AND MORPHOLOGICAL ANALYSIS

Investigative analysis of ES swipes and other media (such as air filters, soil, vegetation, etc.) allows the IAEA to look for particular clues or indications of small amounts of material which may indicate possible undeclared activities, past or present, even in cases where materials and/or facilities are hidden or equipment is cleaned. In particular, the elemental and/or chemical composition of various materials at a location may reveal the presence of NM or key NFC-related materials. Materials of interest may include the following:

- U and Pu compounds (for example Esaka, et.al., 2016 [3], demonstrated the applicability of SEM to the characterization of U-containing particles, and Ranebo, et.al., 2002 [4], demonstrated the applicability of SEM to the characterization of U and Pu-containing particles);
- Cladding materials such as zirconium alloys (consisting of hafnium-free zirconium);
- Special alloys of steel or high-strength aluminium, applicable to centrifuge enrichment;
- Tungsten or tungsten alloys (for example Sheppard, et.al, 2007 [5], showed the capability of SEM techniques in finding and characterizing tungsten particles collected on air filters);
- Other materials in purified form, such as metals or other materials whose origin needs to be clarified (such as anthropogenic vs. naturally-occurring);
- Particulate which has undergone certain types of processing (e.g., manufacturing, heating, melting, etc.) and may reveal certain NFC or industrial processes.

It is expected that an elemental and morphological library covering the materials above will greatly aid evaluators in interpreting signatures collected on ES samples.

2.1 Elemental Analysis

The primary instrument used for the characterization of elemental concentrations in particles is the SEM, with a variety of associated detector and software systems.

2.1.1 Automated Particle Analysis (APA)

Software and hardware systems are available that allow the laboratories to perform APA, allowing to search for and characterize large numbers of particles based on elemental composition, size, etc. APA provides information about the number and types of particles loaded onto a substrate, including their location on the substrate. Subsequent in-depth elemental analysis can be performed on specific particles, which represent particular materials or have specific qualities of interest.

2.2.1 Energy Dispersive X-ray Spectroscopy (EDS)

Elemental analysis is often performed using EDS which has several general characteristics:

- Large numbers of particles can be analysed rather quickly, in particular when combined with APA systems;

- Elements with a wide range of atomic numbers can be characterized (i.e., from light elements such as B, C and F to heavy elements such as actinides);
- Quantification of elemental concentrations may be more difficult (i.e., results may be more qualitative than quantitative). EDS is often used to determine which elements are present and to estimate relative abundances, but not necessarily for accurate elemental composition.

2.2.2 Wavelength-Dispersive Spectroscopy (WDS)

Elemental analysis is often performed using WDS which has several general characteristics:

- Advantageous in resolving spectral interferences (e.g., U-Pu);
- Analysis generally takes longer and fewer particles can be analysed;
- Elements with low atomic number are more difficult or not possible to analyse.

In addition, other techniques such as optical microscopy and micro-Raman spectroscopy may be used to help identify and/or characterize particles made of U compounds, graphite, or other chemical compounds or materials of interest. For example, Pointurier, et.al., 2010 [6], demonstrated the use of micro-Raman for the identification of UO_2, $UO_4\cdot(4H_2O)$, U_3O_8, UO_2F_2, and UF_4.

2.3 Morphological Analysis

Images produced via the SEM instrument provide ES evaluators with information about the size, surface characteristics, homogeneity, possible compound, and/or possible processing history or source of the particulate. Images are commonly produced from either secondary electrons or backscattered electrons, each with their own advantages. Morphological data for a particular sample may indicate characteristics of certain materials that originate from certain compounds of NM and/or important stages of the NFC; in addition, signatures can be compared to images of known materials measured by the IAEA in the past which may have characteristic morphologies (see Section 5).

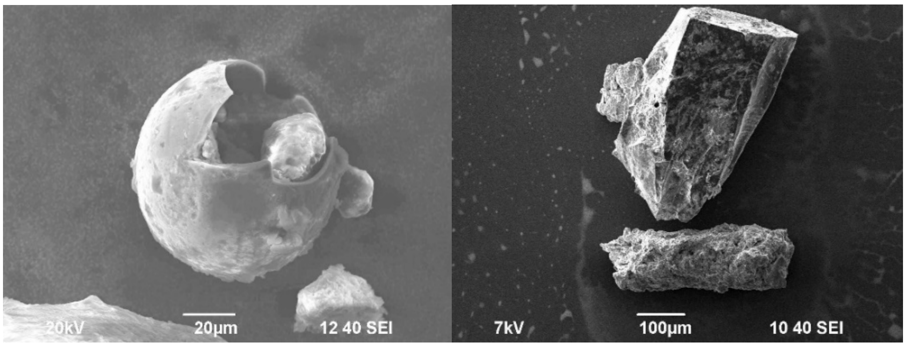


Figure 2: Examples of secondary electron images for particles with unusual morphologies (images courtesy of ESL)

2.3.1 Secondary Electron (SE) Images

SE images are most useful for understanding the topography of a particle's surface and its volumetric morphology. Textures, facets, etc., are generally visible; these characteristics may indicate a specific origin, provenance, or certain release process.

2.3.2 Backscattered Electron (BSE) Images

BSE images provide information about a larger volume of a particle. Therefore, they allow distinguishing particle compositional heterogeneities which may provide evaluators with more information about the origin of certain elements (U for example) in a particle (e.g., if is it uniformly distributed throughout a particle or if it comes from a small inclusion in a particle). An example of SE and BSE images for a calcium fluoride particle with a zirconium-containing inclusion is shown in Figure 3.

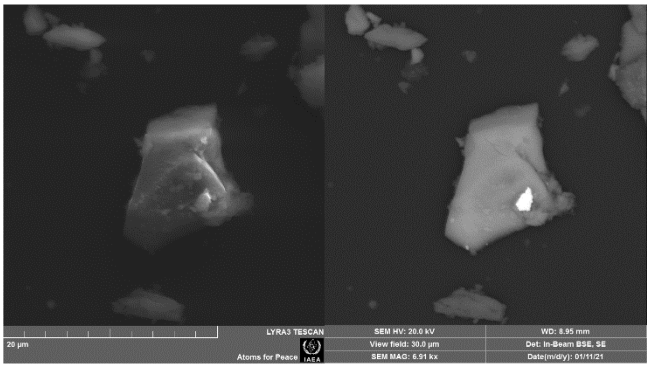


Figure 3: SE image (left) and BSE image (right) for a particle primarily consisting of calcium fluoride with a small zirconium-containing (white color on BSE image) inclusion (images courtesy of ESL)

3. DEVELOPMENT OF AN ELEMENTAL AND MORPHOLOGICAL LIBRARY

The IAEA has developed a library which includes SEM images linked to a database that includes additional information regarding each particle. The images and database form an SEM library that may be updated as needed with new images and data received from laboratories in the future. The library contains the raw images of particles produced by the laboratories with file names linking them to sample collection information. In some cases, multiple images may be available for one particle, e.g. SE and BSE images. As shown in Table 1, the particle database contains pertinent information such as sample and particle number, particle size, elemental composition, type of facility where the sample was collected, etc.

The first library created consists of real images of particles from nuclear facilities, including specific compounds of NM, as well as some other common materials collected on ES swipes. Images were compiled together with particles sizes, elemental and U isotopic composition as well as the type of nuclear activity being conducted where samples were collected. Particles in the IAEA's database can be categorized by sampling location with regard to nuclear or other activity, for example: (1) mining of U ore, (2) U enrichment, (3) fuel fabrication, (4) hot cells of nuclear facilities, etc.

The utility of the SEM library in combination with software depends on the number of images and associated data available. The usefulness of the library will increase over time as the size of the library increases, including the types of materials and NFC processes covered. The library can be compartmentalized into subfolders to allow the user to clearly separate certain sets of data.

Future expansions of the library will include more data that has been historically received by the IAEA from the NWAL. Such data is currently stored in various formats, often requiring manual extraction, sorting and manual data entry. ES mass spectrometry data received by the IAEA follow strict formatting guidelines, which allows automated loading routines and quality control. The IAEA is currently drafting new data requirements in conjunction with the NWAL, and improved formatting guidelines for SEM elemental and morphological data will be included. This will allow new data to be more easily integrated into the library in real time as they are received from the NWAL in the future.

Table 1: Overview of exemplary particle data which is linked to SEM images

Swipe_ID	Particle Number	Elemental String, wt.% (unc.)	Activity	Form	Size_Min, µm	Size_Max, µm	^{235}U, at.%
XXXX-XX-XX	5001	U:65(5); O:30(5); Cu:5(3)	Mining	irregular sharp	15	25	0.72
XXXX-XX-XX	2001	U:60(3); F:32(2); O:4.5(1.2); Al:1.8(0.2); Ca:0.9(0.2)	Enrichment	spherical smooth	1.5	1.5	0.8
XXXX-XX-XX	5005	U:86(4); O:14(3)	Fuel fabrication	irregular smooth flat	5	10	1.9
XXXX-XX-XX	3010	U:68(2); O:22(2); Al:9(1); S:1.5(0.3)	Hot cell	irregular sharp caverns	14	14	0.18

4. DEVELOPMENT OF ELEMENTAL AND MORPHOLOGICAL SOFTWARE

After design and creation of the IAEA's elemental and morphological particle library, the Visual SEM software application was developed to interact with this particle data, making this data more useful to evaluators. Visual SEM was written in the JavaScript language, a high-level and commonly used programming language, and is compatible with the Microsoft Windows® platform used by IAEA evaluators.

The software links sample information, analysis results (e.g., elemental composition, compound type, particle size, characteristics, etc.), and morphological images (SE and BSE) so that the user can view important elemental and morphological characteristics, compare to particles of known materials, NFC process types, and perform searches. Images of particle matches can be exported as individual files or combined into multiple particle figures for direct use in evaluation reports. Elemental charts can be produced and exported. The inputs and outputs of the program are shown in Figure 4.

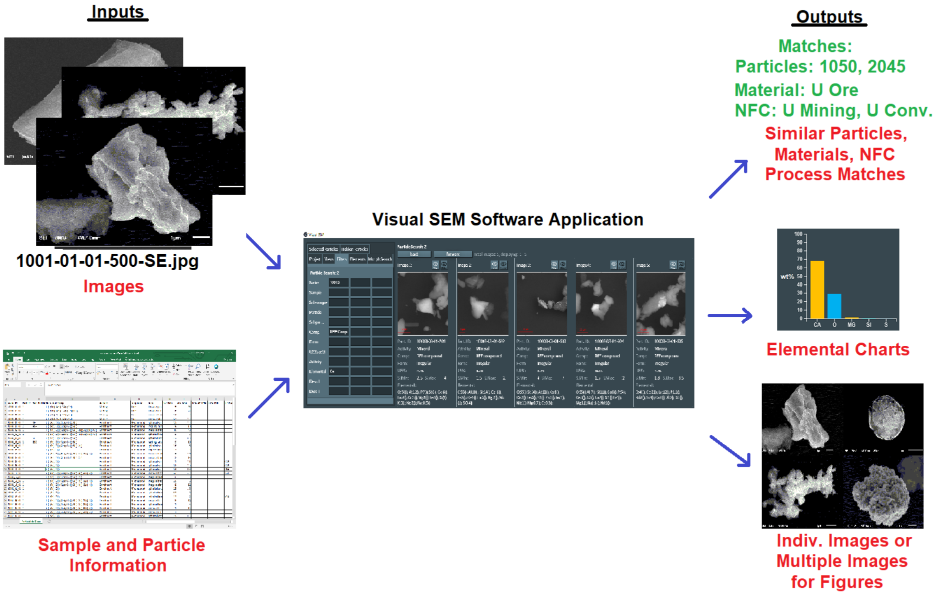


Figure 4: Design of the Visual SEM application

4.1 Functionality

In particular, the software allows the user to easily perform the following functions:

- Import of particle images (including SE and BSE images in *.jpg, *.tif, or *.png format);
- Import of particle characteristic information (MS Excel® *.xlsx format);
- Creation of multiple views corresponding to different materials or sample criteria of interest;
- Filtering by a variety of sample, elemental, particle, and general morphological characteristics;
- Particle hiding, within a view or globally, to help refine datasets;
- Export of images or groups of images for the creation of figures for reports;
- Provides a platform for future automated search based on image characteristics (morphology).

Particle Search: 2
Series: 10003
Sample:
Subsample:
Particle:
Subpart.:
Comp.: REE Compı
Form:
U235 at%:
Activity:
Elemental: Ce
Desc 1:
Desc 2:

Particle Search: 2
Total images: 5, displaying: 1 - 5

Image 1:
Part. ID: 10003-03-01-501
Activity: Mineral
Comp: REE compound
Form: irregular
U5%: n.m.
S.Min: 2.5 S.Max: 4
Elemental:
O:56(); Al:12(); P:7(); Si:6(); Ce:6(); La:4(); Ca:3(); Mg:1(); Fe:1(); S:(1); K:1(); Nd:2(); Na: 0.5()

Image 2:
Part. ID: 10003-03-01-502
Activity: Mineral
Comp: REE compound
Form: irregular
U5%: n.m.
S.Min: 1.5 S.Max: 2
Elemental:
O:55(); Al:10(); Si: 14(); Ca: 6(); Fe:5(); Ce:5(); La:2(); Mg:1(); Nd:(1); S:0.4()

Image 3:
Part. ID: 10003-03-01-503
Activity: Mineral
Comp: REE compound
Form: irregular
U5%: n.m.
S.Min: 4 S.Max: 7
Elemental:
O:55(); Si:14(); Al:11(); Ca:8(); Ce:3(); Fe:3(); S:1(); Zn:1(); La:1(); Nd:1(); Mg:0.7(); Cl:0.3();

Image 4:
Part. ID: 10003-03-01-504
Activity: Mineral
Comp: REE compound
Form: irregular
U5%: n.m.
S.Min: 1.7 S.Max: 2
Elemental:
O:55(); Al:7(); Si:11(); Ca:8(); P:5(); Ce:3(); K:3(); La:2(); S:1(); Fe1(); Mg:1(); Na():1(); Nd:1()

Image 5:
Part. ID: 10003-03-01-505
Activity: Mineral
Comp: REE compound
Form: irregular
U5%: n.m.
S.Min: 1.8 S.Max: 3.5
Elemental:
O:40(); Ce:21(); La:12(); P:11(); Nd:6(); Si:4(); Ca:4(); Al:2(); S:1();

Figure 5: Search and display of particles identified as REE compounds

4.2 Elemental Filtering

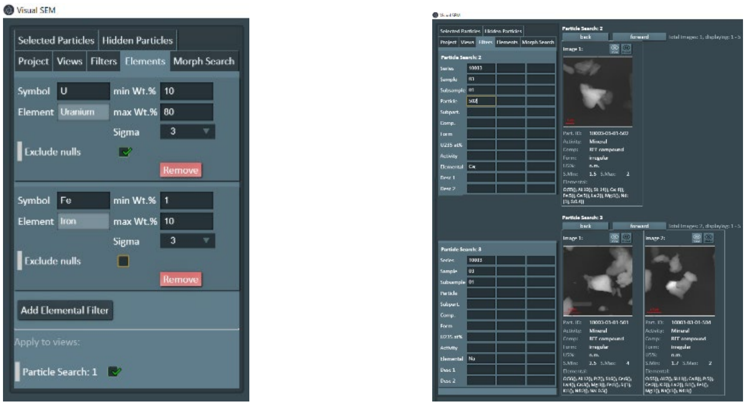
Concerning filtering based on elemental analysis, the software is designed to filter elemental concentrations with or without associated uncertainties (see Figure 6).

4.3 Particle Comparison via Multiple Views

Multiple views allow the evaluator to conduct multiple searches simultaneously (see Figure 6), in order to compare particles of various compounds, elemental compositions, NFC processes, etc.

Typical examples of the use of multiple view include:

- Comparison of a particle of unknown origin to distinct material(s)/compound(s);
- Comparison of particles with unusual morphologies to particles with known processing histories;
- Comparison of particles from different sample collections;
- Comparison of particles with or without certain elemental impurities.



Symbol: U
Element: Uranium
min Wt.%: 10
max Wt.%: 80
Sigma: 3
Exclude nulls ☑

Symbol: Fe
Element: Iron
min Wt.%: 1
max Wt.%: 10
Sigma: 3
Exclude nulls ☐

Apply to views:
Particle Search: 1 ☑

Figure 6: Elemental filtering (left) based on elemental composition (including uncertainties, when provided). Comparison (right) of particles with particular characteristics using multiple views.

4.4 Image Export and Generation of Figures for Reports

Some of the obstacles faced by ES evaluators in the past when using elemental and morphological data have been the lack of an elemental and morphological library, lack of tools for plotting elemental results, lack of search functions, and the time required to find, extract, and/or organize images for inclusion in reports. Visual SEM allows evaluators to readily find and extract images which may be useful for reports, combine numerous particles into ready-to-use figures for evaluation reports, and create and export elemental charts. Images and charts which can be exported include (see also Figure 7):

- Individual Images: Evaluators can find and export an image of a single particle of interest for use in other graphics applications or insertion directly into reports;
- Combined Images: Evaluators can select images of multiple particles and create an image matrix (e.g., 2 x 2, 1 x 3, etc.) which is ready-to-use in evaluation reports, saving time;
- Elemental Charts: Evaluators can generate charts summarizing elemental composition for detected elements or in a “table of elements” format (for “fingerprint” comparison).

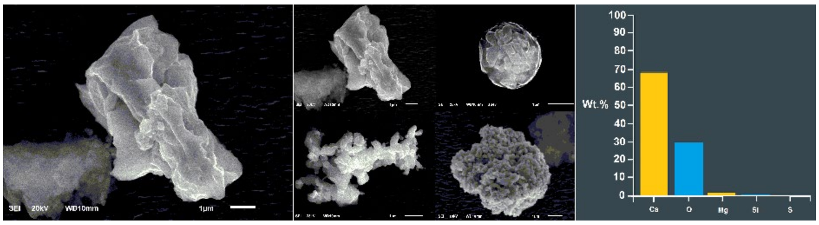


Figure 7: Examples of the export of a single image (left), multiple image matrix (center), and a standard elemental chart (right) – SEM particle images courtesy of ESL

5. AUTOMATED MORPHOLOGICAL ANALYSIS

The dataset used for this study consists of a wide variety of SE particle images. The particles have distinctive morphology such as shapes and textures, for which it is desired to design an algorithm for automated visual image analysis rather than only by text or chemical properties, in order to improve the effectiveness of particle search and comparison. In image processing, the identification or discovery of different visual patterns, such as color, shape, texture, size, brightness, etc. is known as feature extraction. Features can vary over an image and are usually associated with different types of objects in the image. Once features are extracted from a dataset, features can be assigned to different labels by using a machine learning algorithm known as a classifier. When labels are provided by experts as training data, it is known as supervised classification.

Computer vision has evolved dramatically in recent years, moving from hand-designed feature extraction, to data-driven features learned directly from the data using deep learning. The shift from hand-designed features to learned features was made possible by 1) very large image datasets for training algorithms with sufficient variability, 2) development of powerful computing hardware, specifically GPUs, to efficiently implement neural network algorithms, and 3) very deep neural networks that have millions of weight parameters to better learn small differences in image features. Despite the technical advances in deep learning, classical machine learning algorithms are still useful due to their simplicity to train and maintain, so are considered for the Visual SEM tool as well.

The particle image database that has been established thus far has been manually sorted by the team into training data for several potential categories of interest. For the initial data analysis, we treated the provided training category information as generic labels, without using any auxiliary expert knowledge of Safeguards processes or chemistry. The supervised image feature extraction and classification methods we investigated included Gray-level Co-occurrence Matrix (GLCM) features, Random Forest, and pretrained EfficientNet convolutional neural network (CNN) feature layers (see Figures 8 and 9).

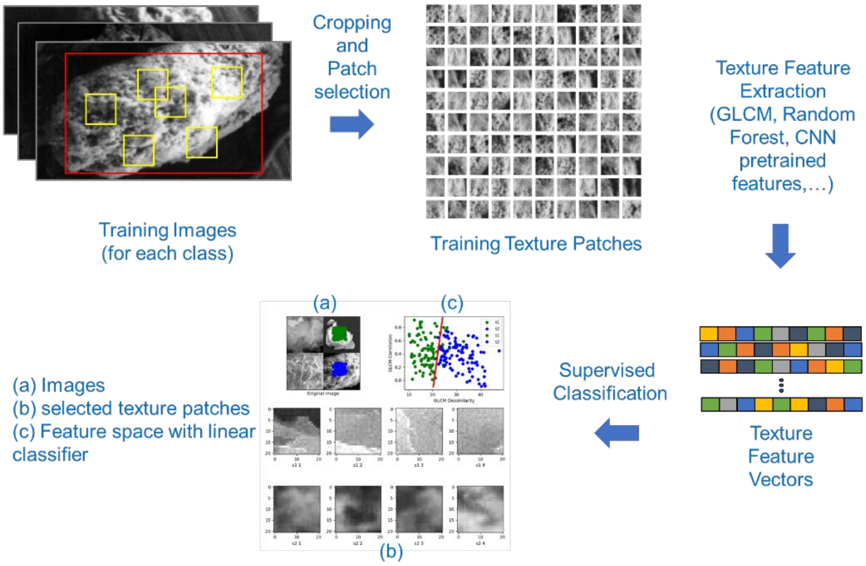


Figure 8: Machine learning workflow for texture classification

The number of available images for each particle class varied from only a few images to tens of images. To create larger training datasets, the images were divided into smaller texture patches ranging from 11 x 11 pixels to 24 x 24 pixels using random 2D patch samples. For our texture classification experiments, at least 50 to 200 texture patches were used for each class, and an equal number of texture patches were used to train each class to avoid class imbalance. The images have wide variability in imaging conditions, SEM imaging method, image format, size, noise, and clutter including embedded text and annotations. Standard image cleaning and pre-processing methods were used to attempt to standardize the dataset. Additional image cleaning algorithms would have to be considered and implemented to properly (and potentially automatically) clean and ingest new images into a large training database prior to training of a chosen classification algorithm on a representative dataset. Image denoising was also implemented to reduce some of the artifacts seen in the images, in order to suppress noise and enhance discriminative textures, but comparison of denoised versus non-denoised images for classifier input is ongoing. The texture features from all methods show promising initial results.

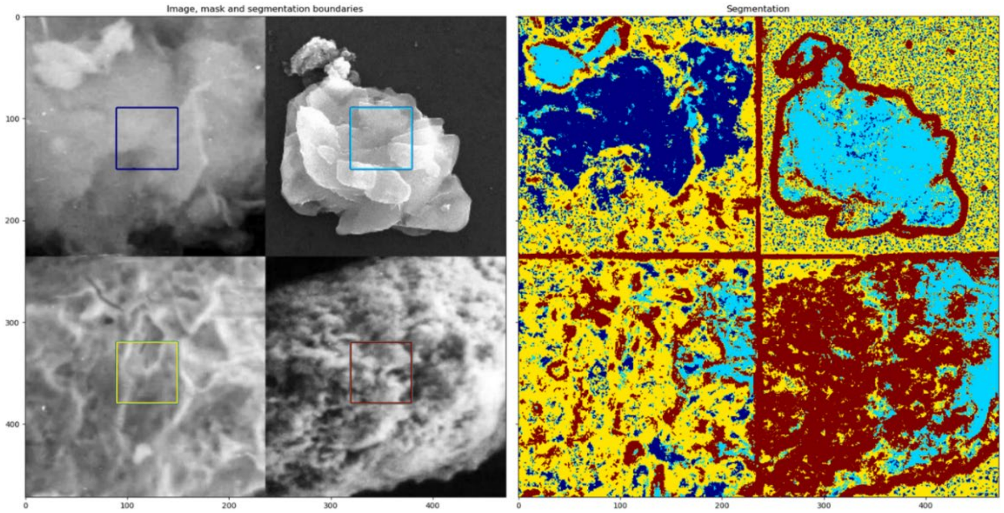


Figure 9: Four particle class examples with Random Forest classification on small texture patches, visualized as image segmentation maps

6. CONCLUSIONS

The challenges facing the IAEA regarding its safeguards verification activities continue to change over time. After the first 25 years of environmental sampling, it is clear that a larger number of samples are taken during sampling activities at locations which require an increasingly investigative approach, involving the characterization of not only NM but also various materials related to the NFC. Techniques such as SEM are crucial for the elemental and morphological characterization of collected particulate.

6.1. Improved Use of Elemental and Morphological Data

The IAEA's new elemental and morphological library makes it possible for evaluators to make better use of historical data to understand key NM and NFC material characteristics and for comparison to new sample collections. The new software described in this paper should serve as an important tool for ES evaluators, help them utilize SEM results, and make more robust SG-related conclusions.

6.2. Recommended Areas of Future Work

Key areas of recommended future work include:

- Continued development of the IAEA particle library and inclusion of more historical data reported to the IAEA in the past. This could also include work with laboratories that have already developed particle libraries of key NM and NFC materials, as well as new analytical requests for archived samples of interest;
- Investigate the availability of commercially-available NFC materials for expansion of the library;
- Continued testing of the software by ES evaluators to learn more about their needs;
- Studies involving the sensitivity of morphological data to various parameters such as elemental impurities, sampling details, process history, etc.;
- Continued work in the area of automated morphology search based on image libraries and implementation of search model(s) in future versions of the software.

7. ACKNOWLEDGMENTS

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