Application of computer image analysis and scanning electron microscopy in environmental engineering and waste management

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Creation and refinement of modern research methods in the last century had a significant impact on the dynamic development of various fields of science. Special attention deserves computer image analysis and scanning electron microscopy. These methods can be used in virtually any field of science dealing with solids. Modern methods have many applications in environmental engineering. Studies, using the above advanced testing methods are also widely used in waste management.

The article presents progressive research methodology of using image analysis and scanning electron microscopy in environmental engineering and waste management. It especially presents the application of these methods to determine physical, chemical and mineralogical properties of solid waste.

Keywords and phrases: scanning electron microscopy, computer image analysis, solid waste, and environmental engineering.

Introduction

Man explores the world by five senses, but most information derives from the sense of sight. Unfortunately, human eye is not able to recognize details of small objects. Therefore, for centuries people were trying to create instruments that would allow them to observe even the smallest objects. As early as the seventeen century first optical microscopes were created. It allowed observing objects in meaningful magnification. It still was not enough for researchers and they strove to create a tool to observe objects on the atomic level. In 1982 the first electron microscope was built. To „light” the object it uses electron beam rather than visible light beam and allows for the observation of single molecules of the object.

With new quality and magnitude of available data automation of data processing became a must. Scientists developed methods for image data processing (computer image analysis). It is used for image processing, analysis and recognize. It is using the analogy with the natural human visual system in its action. Computer operations are trying to imitate the visual perception that occurs in the human brain [1].

In the last decade interest in image analysis and electron microscopy in various fields of science and industry increased. These methods are applied separately and combined in various disciplines such as medicine, biology, mineralogy, or mining and geology. Electron microscopy and computer image analysis can be also used widely in environmental engineering and study of the properties of solid waste. This article will focus latter.

Characteristics of research methods

Scanning electron microscopy (SEM)

Electron microscopy allows for the observation of surface and internal structure of biological objects (e.g., plant and animal), chemical objects (e.g., crystals, polymers), physical objects (e.g., alloys), archaeological objects (e.g., the fossil) and material objects (e.g., cloth, synthetic fibres). Because of the wide range of research opportunities, such as mapping the surface of the object and measuring their chemical composition, electron microscopy is one of the most universal methods of analysis used in environmental engineering.

In the SEM, an electron spot beam bombards the sample. The beam produces an emission of secondary electrons through a linear sweep of the sample's surface. Thus, the sample emits a variety of signals. These signals are recorded by detectors and converted sequentially into an image of the test samples or X-ray spectrum [2]. SEM
consist of an electron column, that creates a beam of electrons; a sample chamber, where the electron beam interacts with the sample; detectors, that monitor a variety of signals resulting from the beam-sample interaction; and a viewing system, that constructs an image from the signal.

There are a few types of signals that generate information about the properties of the samples. These signals can be divided into groups:

- secondary electron emissions gives information about:
  - the topography and morphology of the sample;
  - the crystalline structure of the sample;
  - the distribution of potential and intensity of electric and magnetic fields in the sample;
- backscattered electron emissions provides information about:
  - the topography and morphology of the sample;
  - the distribution of magnetic domains in the sample;
  - the composition contrast;
- Auger electron emissions allows to:
  - chemical analysis of surface layers of the sample;
  - measurements of local potentials in the sample;
- cathodoluminescence provides information about:
  - recombination processes in the material;
  - impurities, additives and structural heterogeneity in the material;
- characteristic X-ray emission allows to qualitative and quantitative chemical analysis of the material. There are four primary measurement techniques:
  - qualitative analysis of the whole picture, which is used to identify the elements in a selected field;
  - element mapping, technique that allows to identify surface elements in a microarea;
  - linear analysis, which shows element distribution along a line;
  - quantitative analysis at a point that determine elements present at a set point and to identify the microstructure [3].

Received images of the same sample surface have a different character depending on the type of signal that is subjected to detection. There are two basic types of these images:

- electron image, which shows inequalities and the geometry of the sample surface;
- X-ray image, which shows what kind of atoms are included in the sample and how they are arranged on the surface of the sample (chemical analysis) [3, 4].

The result of a sample analysis is an image with a material surface and with marked points which can be analyzed, and a map.

Conventional SEM requires samples to be imaged under vacuum, because a gas atmosphere rapidly spreads and attenuates electron beams. Consequently, samples that produce a significant amount of vapours, e.g. wet biological samples or oil-bearing rock need to be either dried or cryogenically frozen. Processes involving phase transitions, such as the drying of adhesives or melting of alloys, liquid transport, chemical reactions, and solid-air-gas systems in general cannot be observed. The environmental scanning electron microscope (ESEM), which was developed in the mid eighties, is a SEM that allows a gaseous environment in the specimen chamber. Its primary advantages lie in permitting the microscopist to vary the sample environment through a range of pressures, temperatures and gas compositions. The ESEM retains all of the performance advantages of a SEM, but removes the high vacuum constraint on the sample environment. Samples may be examined in their natural state without pre-treatment [5].

Computer image analysis (CIA)

An average person has contact only with image analysis techniques related to digital photography. However, the application of specialized software for analysis and processing of images allows the use the computers in various areas of science and life. This method allows the user to accelerate and automate the measurements. Also it provides repeatability and reproducibility of analytical results and access to a large number of measured parameters, which broadens the potential for research.

CIA is a set of methods that allows for the treatment of imaging data. It consists of consecutive operations. Successful implementation of these operations depends on the proper selection of parameters for each operation and determines the final analysis result.

The first step in CIA is digital image acquisition, a number of physical and electrical phenomena associated with the implementation, adaptation and storage of the image, as well as mathematical operations, which results in an artificial representation of the surface in a form of digital image on the screen. Converting the information contained in the image into digital form is convenient for data processing. The basis of the acquisition is to define the parameters of the artificial representation of an image on the computer in appropriate way. These parameters include:

- raster graphics — digital representation of an image is based on a discrete presentation of the image as an array of numbers that describe the colour of individual image points;
- format in which the image is saved after discretization, including:
  - three-dimensional resolution — measure of the ability to recognize the details of the image.
The most common formats are binary (black and white, 1-bit image), monochromatic (256 grey levels, 8-bit image) and colour (RGB, 24-bit image) [6].

The next steps in CIA are image pre-processing operations. They are used to improve the quality of the digital image entered into the computer, correct the errors brought by an operating system and for other additional operations that improve image quality. These operations are divided into:

- Type 0 operations (point operations) — the output intensity level at a certain pixel is strictly dependent on only the input intensity level at that point, frequently used in image segmentation, pixel classification, image summing, differencing, etc.;
- Type 1 operations (local operations) — the output intensity level at a pixel depends on the input intensity levels of the neighbouring pixels as well, e.g.: edge detection, image filtering, etc.;
- Type 2 operations (geometrical operations) — the operations are such that the output level at a point is dependent on some geometrical transformation, e.g.: shifts, rotations, reflections and other transformations of image geometry [7].

An important element used in almost all of the CIA algorithms is binary conversion and segmentation of the image. It is one of the points operations. Binary images are images whose pixels have only two possible intensity values (0 for black and either 1 or 255 for white). These images are often produced by thresholding a greyscale or colour image, in order to separate an object in the image from the background. The colour of the object (usually white) is referred to as the foreground colour. The rest (usually black) is referred to as the background colour. However, depending on the image which is to be threshold, this polarity might be inverted. The well-established segmentation techniques are histogram-based thresholding, region growing, region splitting and merging, clustering/classification, graph theoretic approach, and rule-based or knowledge-driven approach [7]. Examples of binary conversion techniques are binary conversion with the lower threshold, with the upper threshold, with two thresholds, with hysteresis and multi-criteria conversion [3].

The next step of CIA, morphological image processing, implement the most complex operations associated with analysis of the shape of the picture elements and their relative positions. The foundation of morphological image processing is mathematical morphology. Topological and geometrical continuous-space concepts such as size, shape, convexity, connectivity, and geodesic distance, can be characterized by mathematical morphology on both continuous and discrete spaces. Morphological image processing techniques can remove imperfections caused by noise, texture or the inaccurate specification of a threshold and provide information on the form and structure of the image. These techniques typically probe an image with a small shape or template known as a structuring element. This element is positioned at all possible locations in the image and is compared with the corresponding neighbourhood of pixels. Morphological operations differ in how they can carry out comparison. Some test whether the structuring element ‘fits’ within the neighbourhood and others test whether it ‘hits’ or intersects the neighbourhood. The basic operations in morphological image processing are: erosion and dilation, opening and closing, hit and miss transform, and skeletonization [7].

After processing the image is analyzed. Computer system extracts only that information which is relevant for user or process. This implies a significant reduction of data. The results of the CIA are qualitative and quantitative data that describe the specific feature of the image or a whole group of images. Data characteristics depend on the fields of science and technology, in which the analysis was carried out. Two kinds of the parameters are analyzed:

- local parameters, describe averaged, single element of the image, e.g.: the average particle surface, diameter of particles, the average moment of inertia;
- global parameters, describe the characteristics of all elements of the image, they are relative values, which refer to the base parameters, such as surface area or length, e.g.: number of particles per unit area.

On the basis of a set of quantitative parameters, which describe the characteristics of the images, conclusions can be draw [8].

Results and discussion

Applications of scanning electron microscopy in environmental engineering

Biindication

Microscopy is one of the most versatile methods of diagnosing the condition of the elements of the environment. SEM can be used to monitor the environment and determine the extent of its degradation through the rapid and unambiguous diagnosis of the changes in the structure of living organisms. SEM enables researches to create a catalogue of bioindicator species features as comparative material for the diagnosis of the environmental contamination processes. An example of this type
of research could be the qualitative and quantitative analysis of changes occurring on the surface structures of the epidermis of the plant organs (e.g. raid wax). Waxy structures protect plants from harmful external stimuli and the adverse environmental and biological factors; therefore they are good in situ biomarkers of land environmental pollution. Observations in the SEM confirm the high suitability of plant wax in the evaluation of the environment [9].

Biodiversity of living organisms

SEM is useful in monitoring the environment (assessment of the different plant communities, e.g. forest, grassland). Moreover, it is helpful in evaluation of the flora biodiversity by imaging patterns of cell on the plant organs surface. The results of these observations are the microstructure atlases of plants and animals [9].

Applications of scanning electron microscopy in waste management

Determine properties of fine-grained waste

Electron microscopy is widely used to determine the characteristics of the fine-grained waste. In the experiments, SEM is used to determine the morphology and chemical composition of various wastes. Analysis results are often used to specify the suitability of selected waste for its reuse in industry. These researches are also used to determine the stability of waste chemical and geometric composition, quantities of hazardous substances released from waste and products made from waste.

Many researchers were trying to determine the properties of fly ash by using electron microscopy. They tested surface, morphology and chemical composition of the grains. In their studies the authors of this article, determined the elemental, chemical and mineralogical composition of fly ash. Moreover, they tested grain size and morphology of samples. Based on the results, fly ash was classified into different groups. The properties of fly ash were used to determine the most effective methods of its utilization [10–12]. The authors are creating a methodology for testing the properties of this waste using electron microscopy and CIA.

Electron microscopy was also used to determine changes in the properties of fly ash during its weathering [13, 14]. SEM was used to observe changes in the structure of fly ash deposited in landfills. Changes in the morphology and destruction of the fly ash grains was observed under the SEM. Changes in behaviour and appearance of the fly ash stored in landfills indicated that this fly ash have different physical and chemical characteristics then fly ash derived directly from the electrofilters and can no longer be used for the same technical applications.

In the research [15] was explored the possibility of accurate measurements of chemical composition of fluidized fly ash, especially determination of the content of heavy metals and radioactive elements in fly ash. The aim of the study was to find effective ways of fly ash utilization.

SEM can be also used to study the properties of other power industry waste.

It is used to study:

• Waste from semi-dry flue gas desulphurization (morphology and microstructure of grains; the possibility of using waste for the production of sulphite and anhydrite binders) [16],

• Fine-grained waste from the biomass combustion (determining the differences in the construction of particles produced in the fluidized bed combustion of conventional fuels (coal and lignite) and renewable fuels (biomass); dimensional view of microparticles and determine the chemical composition of microparticles) [17].

Hazardous waste testing

Electron microscopy can be also used to study hazardous waste, especially fine-grained waste. This method is used to monitor the waste pollution, to study its properties and to evaluate its effects on living organisms. In addition, it is used to evaluate methods of hazardous waste disposal.

A good example of this type of waste is asbestos. Asbestos is a fibrous silicate mineral that contain hydrated magnesium oxide with admixtures of iron, magnesium, calcium and aluminum. In the seventies of last century, it was found that asbestos causes respiratory diseases because it contains very small dust fibres. Dustiness monitoring is the primary method of obtaining information about the dangers of asbestos. There are methods in which electron microscopy is used to monitor the volume and harmfulness of asbestos fibre dust. There is also the possibility of using a spectral microanalyser to identify the type of asbestos [18, 19].

In addition, electron microscopy is used to evaluate asbestos neutralization technologies [20, 21]. In the paper [20] SEM was used to study the possibility of disposal of asbestos (as well as eternit) in the process of microwave irradiation of asbestos in the presence of phosphate. These studies have shown that the effect of this process is the formation of an amorphous substance, which does not contain any loose fibres with carcinogenic properties.

Waste biodegradation

The structural analysis of polymer waste is another example of the application of SEM. The increase in consumption of synthetic polymers in the packaging industry creates a real threat to the environment. Currently scientist intensively looking for new polymers,
which waste could be disposed in an economic way, for example by biodegradation. SEM allows to evaluate the degradation of post-consumer waste (evaluation of morphological changes of plastics). It also allows to observe the morphology of micro-organisms used in the biodegradation process [9].

Applications of computer image analysis in environmental engineering and waste management

Macroscopic image analysis

Computer image analysis is widely used to define the properties of the waste. Usually the source of the analyzed images are recordings from CCTV cameras and digital cameras. In the waste management and environmental engineering, industrial cameras are used to study the composition, morphology and properties of both municipal and industrial waste.

Digital image analysis can be used to define the structure of municipal waste [22]. In this methodology, computer program in the macroscopic photographic image identifies different groups of waste and determines their surface area. The morphology studies of municipal waste used CIA to improve the counting of waste, and processing of received statistical data.

CIA systems were used also to deal with a material that is very diverse and difficult to identify — scrap [23]. The purpose of this study was to obtain as much as possible information about the composition of this waste, from the charging of scrap into the electric-arc furnace. The information about the scrap is obtained by processing digital images from industrial cameras. The objective of measurement is the correct assignment of scrap to one of several class of scrap in the still plant, and the continuous supervision and registration of data from this process. In addition a database that holds the images of scrap from a digital camera with information regarding the class of scrap metal, its weight, and all information acquired through their processing was created. Database helps select analysis method and image processing parameters, which then allow for automatic classification of scrap during the charging process.

CIA methods were also used in the dismantling of PCB boards [24]. Three-dimensional optical recognition systems is used for the selective dismantling, while for simultaneously dismantling the one-dimensional recognition systems is used. During CIA, an interesting part of the PCB boards are identified by comparing the patterns from the database, and then determine their coordinates and their selective dismantling.

Microscopic image analysis

In the analysis of microscopic images, the sources of the analyzed image are images obtained from optical or electron microscopes. In many cases, electron microscopy and image analysis are used as a combined research methods. There are many examples of applications of these methods in environmental engineering and especially in waste management.

CIA is now very often used to determine the properties of concrete. Nowadays, various kinds of industrial waste are more and more frequently used in the production of concrete (e.g. fly ashes, waste sands). Their use affects on the properties of cement and concrete, therefore, there must develop new and fast test methods for concrete manufactured from waste. Many of these research methods are based on the analysis of microscopic images. In the article [25] CIA was used to characterize the air voids in the concrete as well as to diagnose a variety of aeration in concrete structures. While, in a publication [26] the possibility of using CIA to determine the microstructure and distribution of air voids in the concrete containing fluidized bed fly ash was present. The research has identified total air content, the specific surface area of air voids, and the distribution of these voids in the concrete. Analysis of the results allowed to determine the influence of addition of fluidized bed combustion fly ash on the spacing factor and on the specific surface area of voids in concrete. In the paper [27] CIA and SEM were used in the quantification of the residual mortar content in recycled concrete aggregates. There are significant economic and environmental reasons to use recycled concrete aggregates in making new concrete. The nature and properties of recycled concrete aggregates have a definite impact on the performance of recycled aggregate concrete and the effects can vary considerably. The amount and properties of the residual mortar in the recycled concrete aggregates significantly affect the mechanical and durability properties of the new concrete. In this research CIA was used to quantify the residual mortar content in the different size fractions of the recycled concrete aggregates tested. In the studies [28] CIA techniques were used to characterize the composition of waste-entrained concrete, and to provide quantitative data on the size, shape and location of each component in the samples. Multiple samples were analyzed to determine the degree of heterogeneity in the sample.

CIA is also used to evaluate the properties of other industrial products made from different kinds of waste. An interesting application of this test method is it’s used to study the structure of pellets and briquettes made from fine-grained materials. In paper [29] the sample case of CIA to compare the briquettes structure made on a fine-grained metal material is presented. The pellets were made on the stamp press and the roll press, of wastes from the finishing process of bearing balls and different additional substances as a binder. Based on
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these studies the best combination of amount and type of binder in briquettes was found.

In the paper [30] CIA and SEM was used to study properties of low-cost nullite mullite ceramics produced from bauxite and high-aluminium fly ash. The effects of the doping V$_2$O$_5$ on the bulk density, apparent porosity, bending strength and microstructure of nullite ceramics were studied in detail. This research may provide a new method in utilizing the vast resources of fly-ash waste from power plants in the production of low-cost nullite-based engineering materials.

In the research [31] environmental scanning electron microscope (ESEM) coupled with a digital image analysis (DIA) program (Visilog) were used to estimate the swelling–shrinkage potential (aggregates scale). The swelling clays have been proposed as engineered barriers in geological disposal systems for waste. However, the swelling potential of soils is also considered a prevalent cause of damage to buildings and constructions. For these reasons, it is fundamental to investigate the physicochemical and mechanical behaviour of swelling clays. The CIA combine with SEM is the best method for this research.

Conclusion

Currently, a rapid development of new improved methods is taking place in science. A good examples of this methods are scanning electron microscopy and computer image analysis. SEM is used to observe a surface and internal structure of biological, chemical, physical and material objects at high magnification; while CIA is used to extract, from visual information, this part which is relevant for user or process. Application of this methods allows speeding up the measurements, full automation of the measurements, the reproducibility of results, and giving access to a large number of parameters. Because of the wide range of research opportunities SEM and CIA are one of the most universal methods of analysis used in environmental engineering and especially in waste management.

Based on the above examples of the use of computer image analysis and scanning electron microscopy to study various properties of waste, including hazardous waste, and products manufactured from waste, can be established that this methods simplify and accelerate laboratory tests. These procedures and analysis tools can be used to determine characteristics of the industrial processes and thus can be apply in their control and optimization. As it is shown in the above examples these methods can also be used in the environment studies, as well as in environment pollution monitoring.

Acknowledgements

Article was carried out within statutory work no. 11.11.100.276.

References


