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ADVANTAGES OF APPLICATION OF THE BACKSCATTERED ELECTRON SCANNING IMAGE IN THE DETERMINATION OF SOIL STRUCTURE AND SOIL CONSTITUENTS

J. Wierzchoś[1], *M.T. Garcia-Gonzalez*[2], *C. Ascaso*[2]

[1]Institute of Agrophysics, Polish Academy of Sciences, Doświadczalna 4, 20-236 Lublin, Poland
[2]Centro de Ciencias Medioambientales, CSIC. Serrano 115 dpdo, 28006 Madrid, Spain

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A b s t r a c t. Submicroscopic techniques can be broadly subdivided into the study of micromorphology and imaging of the arrangement of the soil particles and voids, and the microchemical analysis of soil components.

In the authors opinion, one of the most promising tools in the micromorphology study of soil is the Scanning Electron Microscopy (SEM) operating in Backscattered Electron emission mode (BSE) with the auxiliary Energy Dispersive Spectrometry (EDS) as a microanalytical system. The emission of BSE is strongly related to the atomic number of the target. This allows easy localization of resin in pores and soil mineral particles. Qualitative differences of chemical nature between soil constituents are also distinguishable. The SEM-BSE micrographs have a much higher resolution compared with petrographic micrographs, this permits continued observation of soil structure from the meso and micro to the submicro scale. The polished blocks prepared for SEM-BSE study can be simultaneously examined by microanalytical techniques. Highly contrasted SEM-BSE 2-D images can be easily quantified using image analysis systems.

Some examples of application of the BSE imaging in soil micromorphology is given and discussed in the work.

K e y w o r d s: backscattered electron scanning image, soil structure, soil components

INTRODUCTION

There are many physical methods considered to be related to soil structure evaluation. The most common used by soil physicists are the measurements of bulk density, soil textural composition, soil aggregate stability and pore size distribution determined by water retention curves. The point is that quantitative results given by all these methods, tend to be strongly dependent upon the measurement methodology [8]. However, except for soil micromorphologists, the study of soil structure is not usually related to soil structure *per se*, but rather the functionality of soil structure. Taking into account that basic soil structure is size, shape and arrangement of the particles and voids, we can state that soil structure might be considered to be an architectural arrangement of primary particles. These arrangements are mostly restricted to 'pictorial displays' through microphotographs. The observation and evaluation of soil structure using various microscopic methods at various scales provide insights into soil structure and also aid the interpretation of soil formation.

MICROSCOPIC EXAMINATION OF SOIL STRUCTURE

One of the most commonly used microscopic techniques in soil micromorphology is the Light Microscopy (LM). In practice, nearly all thin sections are only studied in transmitted and polarized light. Although, LM contributed sufficient information to the macro scale of observation, the low spatial resolution

did not permit determination of many components and important details of soil particles arrangement. Moreover, LM is not suitable for coordination with a microanalytical apparatus.

Since the work cited of Chen *et al.* [3] many other investigations of soil structure have been realized using Scanning Electron Microscopy apparatus working in secondary electron emission mode (SEM-SE). Although the 3-dimensionality of SEM-SE microphotographs is very helpful in soil micromorphology, unfortunately soil materials do not produce a very high yield of secondary electrons and are a non-conductive substance. Generally these materials must be coated with a layer of heavy metals. But a heavy metal coating renders the sample unsuitable for subsequent analysis with microanalytical systems based on X-ray emission. Moreover, topographic images of the soil surface can contain artifacts resulting from detachment of small particles during sample preparation process and from the obstruction of real arrangement and/or inquires constituents by, e.g., clay coatings. Also the SEM-SE images are very difficult to quantitative description using image analysis systems.

The progress in submicroscopic techniques allows the micromorphological study of soils in submicro scale. Examinations of microporosity [2], mineralogy [20] and organization of soil constituents [13] in that scale were performed by observation of ultrathin sections (usually 90 μm thick) using a Transmission Electron Microscopy apparatus. A great inconvenience of this technique is the difficulty or impossibility of preparing ultrathin sections, because of the heterogeneous density nature of soil material. Another limitation is the small size (1-2 mm^2) of observed area.

Therefore, there is a need of application of other microscopic techniques in the field of soil micromorphology, which permits the observation of wide surfaces of the range of various centimeters as in LM, and giving a high spatial resolution approach to SEM-SE and TEM techniques; permiting the study from meso to submicro range and having the possibility of instantaneous qualitative and quantitative microanalysis *in situ* of the soil features and allowing evaluation of soil structure by image analytical systems.

One of the most promising tool that satisfies the earlier mentioned conditions is the Scanning Electron Microscopy operating in Backscattered Electron (BSE) emission mode with auxiliary Energy Dispersive Spectrometry (EDS) microanaltyical system. The BSE imaging technique promises to be extremely valuable for large areas of micromorphological characterizations of the soils and geological materials from the standpoints of both phase composition and arrangement of features [1,4,7]. Some of the advantages for the morphologist of BSE imaging over SEM-SE imaging were first discussed by Robinson and Nickel [11]. They pointed out that in the BSE image of a flat rock specimen compositional information dominates over the topographic one. The emission of BSE is strongly dependent on the atomic number (z) of the target, which is produced when the composition of the specimen varies over the field of view and arises from the dependence of the backscattered coefficient (η). The coefficient η is simply defined as the ratio of the BSE current to the primary beam current. For pure elements it has been shown [4] that the following empirical expression applies:

$$\eta = -0.0254+0.016z-1.86\cdot10^{-4}z^2+8.3\cdot10^{-7}z^3 \quad (1)$$

For the compounds (most commonly found in soil samples) it is convenient to use the weight mean BSE coefficient ($\overline{\eta}$):

$$\overline{\eta} = \sum_{i=1}^{n} C(i)\,\eta(i) \quad (2)$$

where $C(i)$ is the concentration by weight of each element in the compound, $\eta(i)$ is the elemental BSE coefficient and η the number of elements. This property of BSE allows easy distinction of resin in pores from soil mineral particles. Quantitative differences in chemical nature between soil constituents are also distinguishable with differences of $z>0.1$ and spatial resolution for geological material up to 0.1 μm [6].

Another potential advantage of BSE imaging for non-conductive specimens is that these electrons have high energy and are essentially unaffected by localized charging of the specimen surface, thereby removing the need for surface coating with heavy metals which allows the possibility of applying the EDS microanalytical systems.

The introduction of image analyzing computers to the accurate measurements of pore space and structural units with spatial resolution up to 0.1 μm of the high contrasted and 2-dimensional SEM-BSE images seems to be an important tool in micromorphological description. A number of measurements can be made on features selected for measurements including area, size, shape, perimeter, number, orientation and irregularity. For more detailed data about application of the image analysis to BSE images the reader can find examples in recent works of Smart and Leng [14] and Tovey *et al.* [18,19].

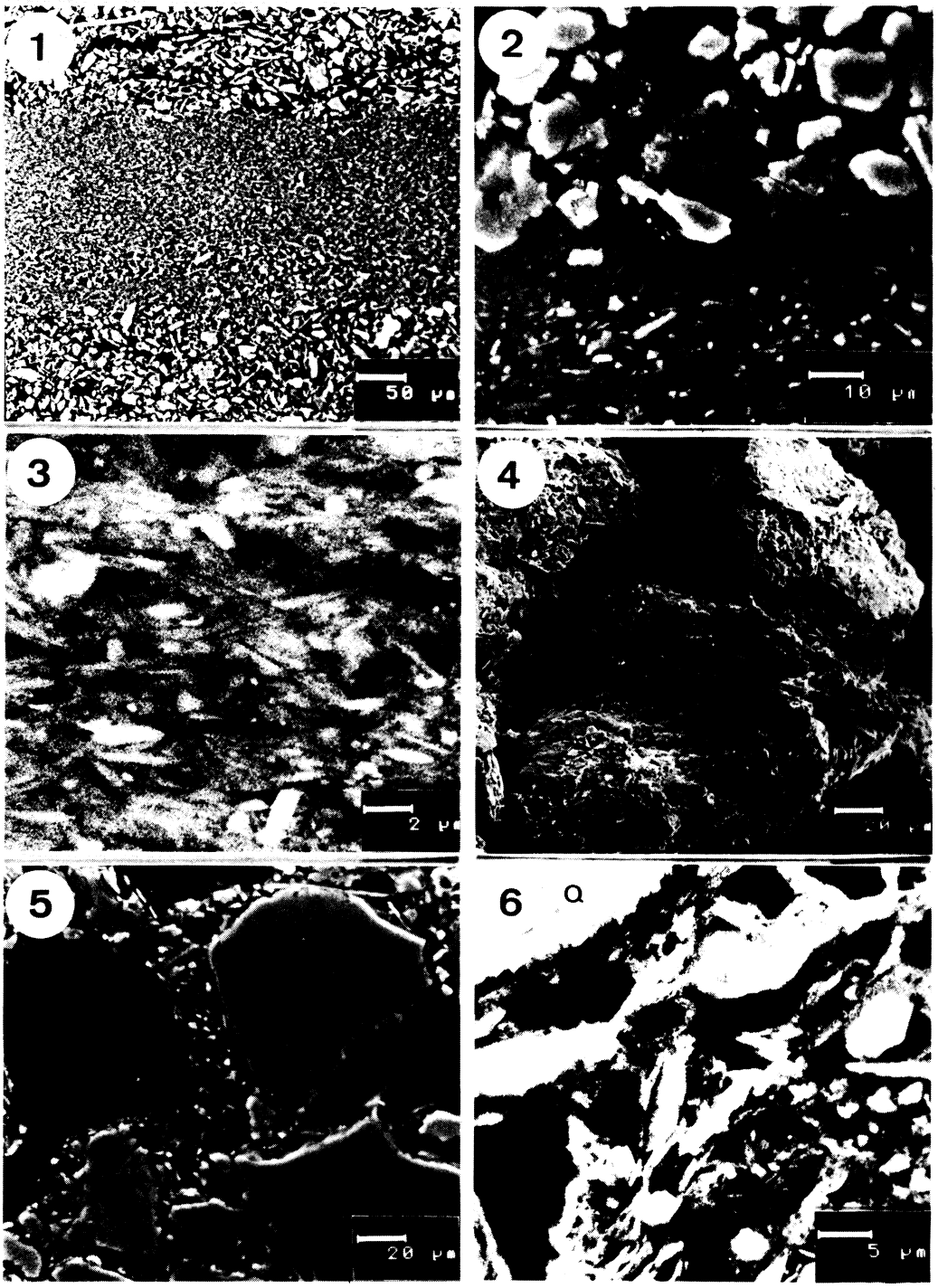
The aim of this work is to improve the knowledge about advantages and possibilities of the application of backscattered scanning electron imaging in soil micromorphology researches.

PREPARATIVE PROCEDURE OF SOIL SAMPLES FOR SEM-BSE EXAMINATION

Considerable attention must be paid to preparation procedure of soil samples using SEM-BSE technique. Soil samples are commonly dehydrated before impregnation. Soils with low clay content can be air or oven dried without shrinkage altering the structure. For clay soils before dehydration soil samples should be equilibrated to the same value of water suction potential and later the most appropriate dehydration method is the solvent replacement by gradual series of acetone [10]. To study the interaction of the root system with soil features the procedure given by Tippkotter *et al.* [17] seems to be the most advantageous. In our study, after the dehydration process the soil samples were impregnated according to Murphy [10]. However, in our opinion the low viscosity resin as Spurr hard resin or LR White medium grade resin should be used to ensure maximum saturation of the micropores system. After polymerization the soil blocks were sawn and the largest side was polished with abrasive powders of decreasing grain size. For the last polishing medium the diamond powder with the grain particles diameter up to 0.1 μm was used. The fine polished surfaces were then coated with an evaporated carbon layer of 50 nm thickness. The samples were then examined with SEM Zeiss 960 DSM equipped with a scintilator type of BSE detector and Link ISIS EDS microanalytical system. Optimum conditions of observation were obtained at 25 kV, accelerating voltage, 8 mm working distance and 10^{-8} A specimen current.

RESULTS AND DISCUSSION

Figure 1 shows a fine laminated fabric of quaternary detric sediments. The presence of this sedimentary material in the Ebro Valley (Spain) is associated with soils that present serious problems with respect to technology, usage and conservation [5]. In the formation of this material two types of layers with different microstructure were distinguished. The layers composed mostly of silt show the skeletal microstructure [12] illustrated in the upper part of Fig. 2. Skeletal pore space is made up of uniformly distributed interaggregate and interparticle open-type pores. Layers composed mostly of clay show a turbulent microstructure [12] as illustrated in the lower part of Figs 2 and 3. Note that BSE images were taken in higher magnification (5000x) to allow the observation of the arrangement of clay particles. It is possible to distinguish that clay micro-aggregates are oriented along the bedding plane. The interaction between the clay microaggregates is of the face-to-face type. The pore space is represented by very small, fissure-like, intermicroaggregate pores. Such a layer can be compared to non-porous crusts which prohibit the passage of roots and act as a physical barrier for water permeability. Presented BSE images (Figs 1, 2 and 3) demonstrate the possibility allowing continued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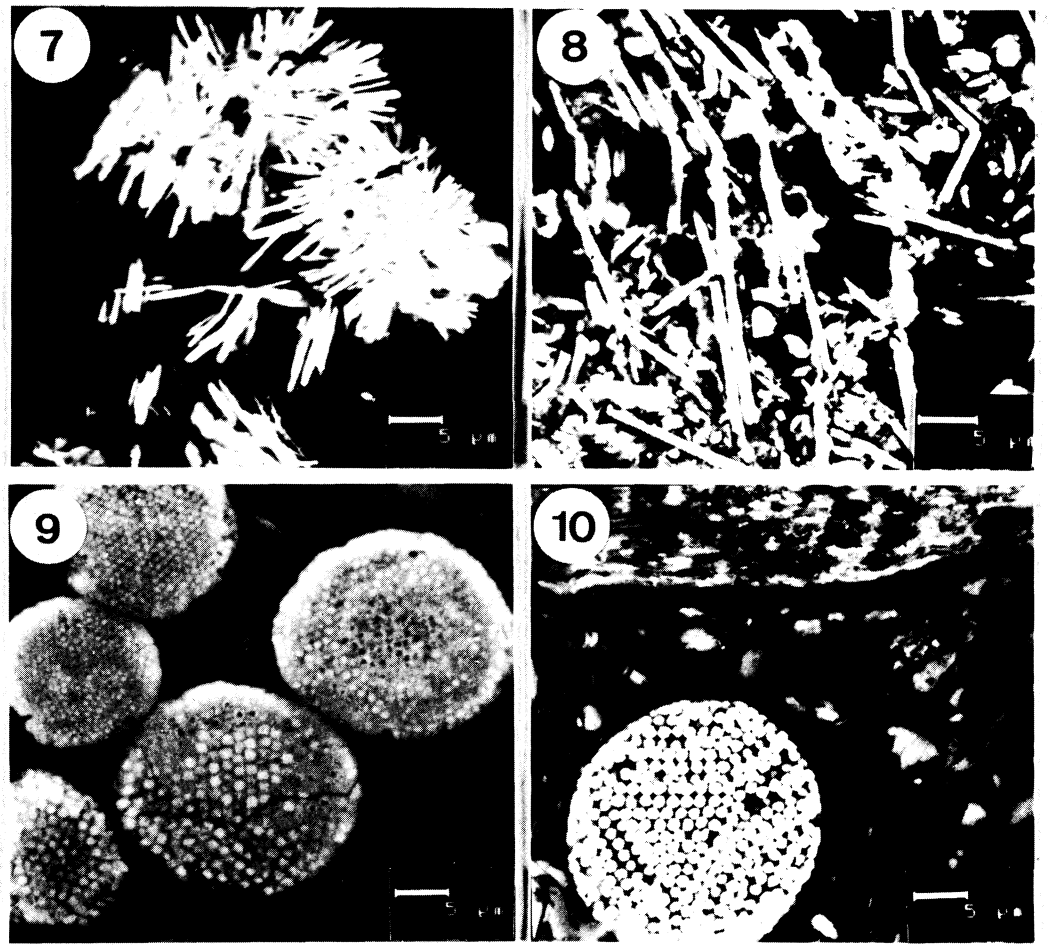

Figs 1-10. 1 and 2 - SEM-BSE images of the fine laminated quaternary detric sediments fabric; 3 - Detail of the lower part of Fig. 2. showed a turbulent microstructure of clay layer; 4 - SEM-SE image of clay coatings; 5 - SEM-BSE image the same soil as in Fig. 4. showed a composition of the granules. Q - quartz, F - feldspar, C - clay coatings; 6 - Detail of the clay particles arrangement within clay coating layer; 7 - SEM-BSE image of the barite microlites; 8 - SEM-BSE image of the acicular crystals of calcite; 9 - SEM-BSE image of the geothite granules; 10 - SEM-BSE image of the pyrite framboide.

study of soil micromorphology from macro to submicro scale.

The micromorphological interpretation of SEM-SE micrographs (Fig. 4) can contribute to several mistakes. It is not clear whether observed granules are massive aggregates or clay coated sand/silt grains. Other difficulties augment the description in detail of the clay particle arrangement along the grain surfaces. The BSE images (Fig. 5) clearly show the interior of the granules composed mostly of silt and sand grains. Figure 6 demonstrates the possibility of the examination of the clay/silt particle organization within the coating layer. Note that clay particles are grouped closely in microaggregates of various sizes with face-to-face contacts dominant. Microaggregates in the form of stepped clusters or chains with oblique edge-to-face contacts create a loose interconnecting framework along the grain surface. The BSE images presented in Figs 4, 5 and 6 were taken from soil classified as Xeric Torriorthents in the U.S.

system [15] and proceeded from the Flumen-Monegros area (Spain).

The satisfactory spatial resolution of the BSE images can improve the knowledge about arrangement of clay particles in the clay coating layers. It can help to distinguish the major associated structures between cutans and other coating features.

The microanalytical determination using the EDS technique performed on the fine polished soil blocks guarantees the best qualitative and quantitative results because the lowest noise/signal ratio of this analysis can be obtained on flat surfaces. In this work we gave some choice examples of EDS microanalysis of soil constituents using as samples the fine polished blicks.

Figure 7 shows BSE images of the barite ($BaSO_4$) microlites found in poorly drained Calcic Xerochrept [15] soil (Valle Ebro, Spain). The evidence of authogenic barite microlites in subarid soils has been only reported twice by Lynn *et al.* [9] and Stoops and Zavaleta [16]. The barite found by us occurring in needle-like shape of the 10 μm long microcrystals were probably formed from saline ground water and the element barium could have been derived from acid igneous pebbles after alteration of the latter. Nevertheless, more study must be undertaken on the evidence of barite in subarid soils.

Figure 8 shows the acicular crystals of calcite ($CaCO_3$). It is a special variety of calcite, viz. a needle-shaped form. The needles consist of more-or-less equidimensional calcite crystals of an indeterminate morphology. This form of calcite was found in subarid Calcic Xerochrept [15] soil (Valle Ebro, Spain) and occurred in pores.

Figure 9 demonstrates the geothite (α FeO (OH)) granules agrupation of diameter 15-25 μm found in waterlogged finely laminated detric sediment (Flumen-Monegros, Spain). Note the honeycomb-like morphology of the interior of the granules.

The BSE image presented in Fig. 10 shows a framboid, e.g., a spherical aggregate of the pyrite (FeS_2) found in siltatation material of the coconut fiber enveloping of the PVC drain. The hexagonal form of the particular submicroscopic crystal was observed. In the upper part of micrographs the altered sheets of biotite were observed.

CONCLUSIONS

The results presented in this paper show that scanning electron microscopy using backscattered electron signal can be extremely important in investigation of soil structure and determination of soil constituents. This technique of direct observation has clear advantages over other techniques such as light microscopy, scanning electron microscopy in secondary emission mode and transmission electron microscopy. One of the principal advantages of backscattered electron scanning imaging is the possibility of the continued observation from macro to submicro scale. The BSE imaging reveals important soil features not apparent or barely detectable using light optical methods. Qualitative and quantitative chemical composition can be assessed by using an energy dispersive spectrometer system coupled with the SEM.

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