

EVALUATION OF THE PORE STRUCTURE OF SOIL SPECIMENS

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ABSTRACT

The pore structure exercises both a direct and an indirect influence on the development of other properties of the soil, in particular on the processes of air and water flow and the ability of the soil to retain water. An attempt has been made at a quantitative evaluation of two basic parameters: the volume fraction and the relative surface of the pores in the soil samples. Evaluation of those parameters was based on the method of stereological analysis and was performed on polished sections. The preparation of the sections was possible after hardening the liquid binder with which the pore spaces of the samples were filled. A method of optical contrasting of the sections was developed for the purpose of easier distinction between the pores (filled with the binder) and the soil particles. Optimal working conditions for the automatic image analyser Metapericolor have been selected so as to ensure correct identification of the components. The results of planar automatic analysis have been verified by comparing them with the results of point and linear analysis carried out directly by the observer.

Keywords: soil structure, contrasting of sections, stereological analysis.

INTRODUCTION

Mechanical tillage of soil is frequently defined as soil manipulation aimed at improving its physical properties and thus ensuring better crop yields. The application of tillage measures causes a number of interrelated processes affecting the soil environment. Volumetric strain results in changes in soil density and porosity, and therefore also in the processes of air and water flow. (Walczak 1977,

Konstankiewicz 1979, Tijink 1988).

In the course of soil strain its structure is the primary object of changes. In soil science there is a number of definitions of soil structure, but all of them define it as a spatial system of solid phase elements and soil pores (Słowińska-Jurkiewicz 1991).

Microscopic observations of soil structure, and especially of its changes under the effect of various tillage measures, indicate that external forces can cause changes in the size and shape of soil aggregates, their decay and crumbling, formation of whole high density blocks of aggregates composed of smaller aggregates, and that the pores filling the solid phase cannot be compared to capillaries or to other known geometrical shapes (Gliński et al., 1991).

The study presented herein was aimed at demonstrating how the method of stereological analysis can be applied in studies on pore structure of soils, through methodological problems involved in the preparation of sections, selection of microscopic image parameters, and the stereological analysis itself. A more extensive study of this problem is at present in print (Bodziony et.al.1993).

PREPARATION OF SOIL POLISHED SECTIONS

For the study a soil was selected for which a complete agrophysical characterization had been worked out (Gliński et al.1991). It was a loess soil (Orthic Luvisol) from a field after potato(Z) and maize(K) crop. Samples of the soil in natural state were taken into test tubes, 22mm in diameter, from Kopecky's cylinders. Further in the study the samples are referred so as series Z(Z1, Z2) and series K(K3, K4, K5, K6).

Soil samples sealed in a binder were prepared on a stand equipped with a vacuum chamber and an autoclave. All the samples were placed in a vacuum chamber on a carousel allowing for successive filling up the samples by liquid binder. After 24 hours in vacuum, the samples were flooded with methyl methacrylate monomer with hardening. The liquid binder wetted the soil very well. After filling the chamber with air the samples were placed in the autoclave where the binder was subjected to a 24-hour process of polymerization in nitrogen at a pressure of 7.5 MPa and a temperature 338 K.

The preparation of soil polished sections involved the following operations: cutting the soil samples into slices about 6 mm in thickness, grinding and polishing one of the flat surfaces of each of the slices. Observation of the soil samples in an optical microscope showed that the pores were filled with the binder to the full. This is evidence that the system of pores in the soil represents a fully open type of structure. However, the optical contrast between the binder and the soil particles was not sufficient for these components to be distinguished with absolute accuracy.

Problems were encountered in developing a method for soil section contrasting. The best results were obtained through curing the soil particles in hydrofluoric acid vapor. Protracted washing and weathering of the soil sections was an important element of the procedure.

Microscopy of cured and non-cured soil section leads to the following conclusions. The binder, or to be more accurate the soil pores filled with the binder, appears to be much brighter and whiter in cured sections. Vapor of hydrofluoric acid affect soil particles, leaving the binder intact. The reflexive capacity of the soil has been diminished.

Magnification ratio of 10x10 was found to be optimal for the best distinction between the two section components. This magnification ensures good visibility of areas covered by the binder or by the soil particles as well as the lines of contact between the two components. For the purposes of stereological analysis only the 10x lens was used. Fig.1 shows the photograph of one of the fields of section Z1, and Fig.2 the printout of the same field after the processing of information by the automatic

image analyser "Metapericolor".

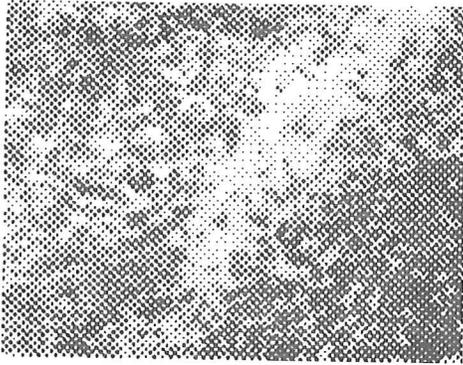


Fig. 1. Soil structure on section Z.1.

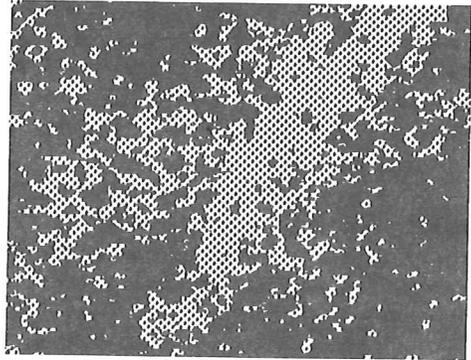


Fig. 2. Identification of structure on section Z.1, performed by the image analyser. Channel III (B).

STEREOLOGICAL ANALYSIS

From among the parameters characterizing the pore structure of the soil there were selected the porosity $\bar{\epsilon}$ and the specific surface area of the pores \bar{S}_V . We shall use the word "porosity" in the meaning of the volumetric content of pores. This is a certain simplification, because porosity denotes the structure of pores, as well. For the purpose of estimation of the porosity the estimators for point and planar stereological analysis were used:

$$\bar{\epsilon} = \frac{n}{N} = P_P \quad (a) \quad \bar{\epsilon} = A_A \quad (b) \quad (1)$$

where:

n - number of points, hitting the pores, from among N -points of the regular grid.

A_A - surface area of the pores per unit area of the section.

For the purpose of estimation of the specific surface area of pores the estimators of the linear and planar stereological analysis were used:

$$\bar{S}_V = 4N_L \quad (a) \quad \bar{S}_V = \frac{4}{\pi} L_A \quad (b) \quad (2)$$

where:

N_L - number of intersections of the traces of the contact surface between the pores and the soil particles per unit length of the measurement line

L_A - length of the perimeters of traces of the contact surface between the pores and the soil particles per unit area of the section.

Estimation of the porosity was performed by means of point and planar stereological analysis of sections in the series Z and K. The point analysis was performed on a semiautomatic stand composed of an Axioplan microscope, an XYZ coordinate meter of a minimum step of 0,625 μm , and

an IBM computer. The coordinate meter, controlled from the computer keyboard, played the role of the microscope stage. It allowed for the realization of counting in the point grid required. The microscope image, in which a 10x lens was used, was transmitted onto a special monitor with cross hairs superimposed on the screen. In the point analysis the primary role was played by the element of personal decision by the observer watching the area surrounding the cross hairs. The selected set of points was used to realize a test of repeatability of countings by particular observers. Optical contrast between the soil components was so clear that individual errors made by particular observers were considered negligible.

From each soil sample one section was taken for examination, and therefore the sections were marked according to the marking of the soil samples. On each section 16 measurement fields were located. These were squares of 1mm side. A statistical analysis was performed for the selection of the grid size in the analyzed field. It was decided to adopt a square grid of size 40 μm , which gives $N=676$ points within a single field.

The main problem encountered in the planar analysis was such a selection of external conditions (illumination, the microscope magnification, possible use of filters) and steps of the automatic processing of images that the final effect, i.e. in our case, the identification of pores and determination of porosity, could be in statistical agreement with the result of the point analysis. In particular, it was decided to read colour images into the computer memory. A colour image is coded by means of three component images (R, G, B), equivalent to shades of grey for the following colours: red, green and blue.

Porosity was determined for each of these three images by means of an automatic procedure. The mean porosity of these three results was also determined using the automatic procedure.

Stereological analysis was carried out in two stages. The aim of the preliminary point and planar analyses, performed on four fields of a section Z.1 was to establish such operation conditions of the analyser that the results obtained conformed to the results of point analysis for the same measurement fields. The preliminary analysis for was performed for 4 fields of section Z.1. The results are presented in Table 1.

Table 1. Results of preliminary point and planar(automatic) stereological analyses. Section Z.1.

Field No	Point Analysis			Planar Analysis	Deviation $ \bar{\epsilon} - \bar{\bar{\epsilon}} $
	Number of points		Porosity $\bar{\epsilon}$	Porosity $\bar{\bar{\epsilon}}$	
	n	N			
1	2	3	4	5	6
1	201	676	0.297	0.326	0.029
2	196	677	0.290	0.317	0.027
3	204	676	0.302	0.310	0.008
4	210	678	0.310	0.305	0.005
Total	807	2707	0.300	0.315	0.017

The principal stereological analysis was performed for all six sections. For control purposes, point analysis was performed for field No 16 of each of the sections. A comparison of the results is presented in Table 2.

Table 2. Comparison of porosity determined by point and planar stereological analyses.

Section No.	Z.1	Z.2	K.3	K.4	K.5	K.6
1	2	3	4	5	6	7
Point Analysis	0.317	0.315	0.312	0.317	0.358	0.411
Planar Analysis	0.316	0.316	0.309	0.311	0.355	0.429
$ \bar{\varepsilon} - \bar{\varepsilon} $	0.001	0.001	0.003	0.006	0.003	0.018

The specific surface area of the pores was determined by means of an automatic image analyzer as another, separate measurement in the course of the porosity assessment. In view of the strenuousness of the linear stereological analysis conducted directly by the observer on a semi-automatic stand, this analysis was carried out only on Z.1 section. The system of parallel measurement lines was composed of 19 segments, each 3mm long, arranged every 0, 5mm. The total length of the measurement lines was 57mm. Table 3 lists the results of the planar and linear analyses. The relative deviation of the specific surface area for section Z.1 amounts to 3.2%.

Table 3. Determination of the specific surface areas of the pores [1/mm].

Section No.	Z.1	Z.2	K.3	K.4	K.5	K.6
1	2	3	4	5	6	7
Point Analysis	72.21	-	-	-	-	-
Planar Analysis	74.60	77.34	76.71	77.24	75.65	76.70
$ \bar{S}_v - \bar{S}_v $	2.39	-	-	-	-	-

CONCLUSIONS

A genuine element of the research study appears to be the developed method of preparing soil samples fixed with a liquid binder and, in particular, the worked out method of contrasting the polished sections of soil. It would be recommended to test the possibility of applying the developed method of contrasting the sections for other types of soil. It would be also useful to adjust this method for microscope objectives of greater magnifications, e.g. 20x, 50x etc. Then, the fractal character of pore structure should be taken into account. The results of point (semi-automatic) stereological analysis were the basis for the estimation of the results of planar (automatic) analysis of the porosity of soil samples. A good agreement between the planar analysis and the results of point analysis allows to state that the automatic analysis of porosity may be applied on a larger scale, however, spot check point analyses are necessary.

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