

Soil & Tillage Research 49 (1998) 249-253



X-ray microtomography to investigate thin layers of soil clod

A. Macedo*, S. Crestana, C.M.P. Vaz

Embrapa, Agricultural Instrumentation, PO Box 741, 13560 970 São Paulo SP, Brazil

Received 12 May 1998; accepted 1 September 1998

Abstract

In this paper we present an equipment and a methodology for soil non-invasive investigation at the microscale. We developed a micrometric scale tomograph to work at a resolution of at least 100 μ m. A microtomography of 1 mm sand grains and 1 mm grass roots is presented. Inside one of the grains, a crack 110 μ m wide per 460 μ m long can be observed. Pores of 100 μ m can be studied. A microtomography of a little soil clod is shown, in which grains of densities up to 4.5 g cm⁻³ can be seen. Pores ranging from 200 to 800 μ m can also be detected in the tomography. As an example of the potential of the method and equipment, soil crusting and sealing can be observed in a natural sample in soil investigations. The image shows three layers of different densities and soil textures. The thicknesses of the layers from top to bottom are, respectively, 1000, 500 and 1700 μ m. © 1998 Elsevier Science B.V. All rights reserved.

Keywords: Microtomography; Soil; Pores; X-ray; Instrumentation

1. Introduction

Soil scientists are trying during the last years to look inside the soil in order to find answers related to water and solute travelling paths, root development, soil crusting and other phenomena. Among these questions mention can be done to those posed by Cassel and Nielsen (1994): "Would there ever be a method to allow us,..., to look inside a soil system to observe individual pores, how they vary in size, how regular or irregular they are, how they are arranged and interconnected, which ones are filled with water, and which ones are actively involved in the water and solute transport processes?" and "Would we ever be able to discern different rates of water and solute transport through different pores?" Techniques like neutron probe moderation, X-and γ -ray transmission, tracer labelling and mercury intrusion, despite their great contribution to soil global parameter measurement, cannot accomplish the mentioned investigations needed in advanced soil research. The development of new advanced and non-invasive techniques is requested.

Computerized Tomography (CT) of X-rays, gamma-rays and NMR are among the well-accepted techniques because of non-invasibility and also of the achieved resolutions. Good results have also been obtained with NMR tomography (Posadas et al., 1996). This technique is, however, limited when investigating soils presenting paramagnetic components or when quantification is of great importance. It is very useful when investigating only the qualitative aspects of non-ferrous materials. X-ray and gammaray CT do not have these limitations.

^{*}Fax: +55-16-272-5958; e-mail: alvaro@cnpdia.embrapa.br

^{0167-1987/98/}\$ – see front matter O 1998 Elsevier Science B.V. All rights reserved. PII: S0167-1987(98)00180-9

Although X-ray and gamma-ray CT have greatly contributed to soil investigation, their results have been achieved in the millimetric scale (Crestana and Vaz, 1998). A few tomographies were accomplished in the micrometric scale, with expensive, not easily available equipment, and with disturbed samples. Among them we can refer to Cesareo et al. (1993, 1994a). Since it is important to investigate not only macro properties of the soil but also to inspect aggregates, grains and pores, a tomograph was developed at Embrapa Agricultural Instrumentation with a resolution of less than 100 μ m.

X-ray and gamma-ray CT are techniques based on the principle of eletromagnetic radiation attenuation by matter. When a beam of rays crosses a sample its number of photons in the direction of the beam can be attenuated following the Beer–Lambert law:

$$I = I_0 \exp(-\mu x),\tag{1}$$

where I_0 and I are, respectively, the incident and the emerging beam number of photons, μ the linear attenuation coefficient of the specific radiation in use and x the thickness of the sample crossed by the beam. The linear attenuation coefficient is the probability per unit length of a photon to be absorbed while crossing the sample.

In X- and gamma-ray CT, several measurements of photon numbers are made in various directions, at chosen angles, known as angular steps. In each direction, a scanning is made in positions separated by a constant distance called linear step. This process is repeated until a scan of 180° is completed. Data so acquired are processed with the aid of a reconstruction software. The detailed reconstruction algorithm can be seen in Rosenfeld and Kak (1982), among others.

2. Material and methods

The equipment here constructed is a first generation tomograph (a narrow beam and one detector only). When the collimators have a geometry such that the beam spreads out instead of being narrow, we can have second, third and fourth generation tomographs. In this case several detectors are used to count the beam photon numbers. Information about the construction of this equipment can be found in Macedo and Crestana (1998). The tomograph was calibrated in terms of linear attenuation coefficients μ and tomographic units (TU) using the method proposed by Crestana et al. (1985). The linear attenuation coefficient calibrated for the energy of 58.5 keV is shown in Eq. (2) (Macedo et al., 1998):

$$\mu = 0.9697 \,\mathrm{TU} + 0.0036. \tag{2}$$

Another calibration can be made in order to quantify the images in terms of densities. In millimetric tomographies this can be achieved using soils with known global densities and correlating them with values of μ measured by the direct transmission method (Crestana et al., 1985). Because of the high resolution in microtomography, this procedure can be used only when examining homogeneous samples. Still another calibration was developed for cases presenting high local densities (Macedo et al., 1998), using the minerals that compose the soil being studied. The following equation was obtained correlating the soil particle density (ρ_s) with μ :

$$\rho_s = 0.5907\mu + 2.2097. \tag{3}$$

Due to the need of a high collimation, a very fluent radiation source is necessary, and therefore, an X-ray system was chosen. The chosen X-ray tube has a fluency of about 10^3 times a 1.1×10^9 Bq Cs137 source.

In the validation phase of the tomograph, homogeneous samples were used, composed of acrylic and epoxy resin. In an intermediate phase, disturbed and homogeneously packed soil were used, and in the final phase, non-disturbed soil clods were employed. One example of a surface sealed soil sample will be shown having 32% clay, 14% silt and 54% sand, previously irrigated and dried in cycles of 1 h irrigation and 24 h drying¹.

3. Results

Fig. 1 shows a microtomography of a sample composed of 1 mm sand grains and 1 mm grass roots. The roots are encircled to emphasise their positions. Due to the total time of acquisition (62 h) they lost part of

¹Sample prepared and supplied by Flávui de Oliveira Silva, from Campinas University – Agriculture Engineering College.



Fig. 1. Microtomography of a sample composed with 1 mm sand grains and 1 mm diameter roots. Pores of $100\,\mu m$ can be noticed.

their water and since the fibres do not attenuate the radiation so much, their presence is not so evident. Pores with dimensions near 100 μ m or less can clearly be seen. One of the grains presents an inner crack, about 110 μ m wide and 460 μ m length. The tomography parameters were a 58.5 keV energy, a 60 mm collimator, an acquisition time of 10 s per point and a beam of 42 000 photons.

In Fig. 2 a microtomography of a surface sealed soil sample is presented. The sealing and crusting phenomena take place in soils due to the impact of water droplets, which de-aggregates the soil and causes the

formation of fine particles. This process can lead to an increase in water runoff during rainfalls or irrigations, and as a consequence, soil erosion may take place. There is still no final agreement of the effects of soil sealing and crusting, and investigations continue to be carried out to determine the causes and influences of these processes on infiltration and erosion (Moore and Singer, 1990; Rawls et al., 1990; Slattery and Bryan, 1994; Fattah and Upadhyaya, 1996; Morin and Van Winkel, 1996)

The image of Fig. 2 presents three layers, the upper being 1 mm thick, the intermediate 0.5 mm thick and the lower 1.7 mm. Fig. 2(b) shows the average linear attenuation coefficients per region. For the energy of 58.5 keV, utilised in this tomography, the Compton effect is prevailing and there is a linear correspondence between the density and the linear attenuation coefficient (Cesareo et al., 1994b). Observing the values in Fig. 2(b) one can state that the intermediate laver has an average density of about 2.1 times the value of the upper one and that the ratio between density averages of the lower and the upper layers is about 1.2. Individual particles can have densities up to 4.5 g cm^{-3} , according to the values observed in Fig. 3 and converted by Eq. (3). The macropores distribution can be visually estimated as being 2-3 times greater in the lower layer as compared to the other ones. The collimator used in this tomography was the same one used and mentioned before, the acquisition time was 8 s per point and the beam 32000 photons.



Fig. 2. Microtomography of a soil sample presenting surface sealing in which one can see: (a) the thicknesses of the three layers formed; (b) the average tomographic units in the indicated regions.



Fig. 3. Variation of tomographic units in transcept L of Fig. 2(a).

4. Discussion

These being among the first results published, microtomograpy can be previewed as a powerful alternative tool to the soil investigate. Details like the ones shown in the surface sealed soil sample has never been investigated in a non-invasive way. No tool until now had this capability. Indeed similar results were obtained with miscroscopy when examining soil surface sealing. But it is an invasive method. The sample must be impregnated with resin and machined. Both this processes can alter the aggregate distribution and pore topology in the micrometric scale level. Thus it cannot be assured that the image is exactly showing the pore geometry the way it was before the sample was disturbed. Associating the microtomography non-invasibility to other techniques, it will be possible to obtain results so long expected and avoid solutions like porosimetry, which face problems of air bubble trapping, volume swelling or shrinking when using water, aggregate damage when using non-wetting liquids, like Hg (Kutilek and Nielsen, 1994).

An important characteristic of microtomography is the capability of evaluating local variations of sample density while millimetric tomographies can only show average values. Fig. 3 shows the variation of μ in transcept *L* of Fig. 2(a), and as stated before, this parameter linearly corresponds to the density.

It must also be mentioned that the tomograph here developed has its application focused on static soil physical parameters or phenomena, due to the total acquisition times, which are of the order of 50 h. An enhancement of this equipment to a third generation tomograph that will shorten acquisition times is desirable and it is among our plannings.

5. Conclusion

Microtomography is an emerging tool that will allow to accomplish new results and insights of opaque and porous systems. It can, for instance, to bring an advance in the study of soil samples presenting surface sealing. The images obtained with this technique showed details that have never before been accomplished with non-invasive techniques. It was possible to show the formation of layers without the need of impregnating the sample. This can avoid aggregate damage, the mischaracterisation of the soil pore topology, the air bubble trapping, like Hg porosimetry, and so on. The results presented here show that it is reasonable to state that the questions posed by Cassel and Nielsen (1994) are close to be answered.

Acknowledgements

We would like to thank Dr. Miroslav Kutílek, from Czech Republic, who encouraged us in this development and helped us in the revision of this paper; the reviewers that greatly helped us to improve the quality of this paper and to clarify the text to the readers; and Embrapa Agricultural Instrumentation (project no. 12.0.94.093) and FAPESP – São Paulo State Research Support Foundation (process no. 90/3773-7) for the financial and logistical support to this development.

References

- Cassel, D.K., Nielsen, D.R., 1994. Introduction: the realization of a dream. In: Anderson, S.H., Hopmans, J.W. (Eds.), Tomography of Soil–Water–Root Processes. Soil Science Society of Agronomy, Madison, pp. 1–5.
- Cesareo, R., Appoloni, C.R., Brunetti, A., Castellano, A., Cruvinel, P.E., Mascarenhas, S., Assis, J.T., Gigante, G.E., 1994a. Industrial applications of tomography and microtomography. In: International Symposium on Computerized Tomography for Industrial Applications, Berlin, pp. 295–302.
- Cesareo, R., Assis, J.T., Crestana, S., 1994b. Attenuation coefficients and tomographic measurements for soil in the energy range 10–300 keV. Appl. Radiation Isot., UK 45, 613–620.
- Cesareo, R., Crestana, S., Mascarenhas, S., 1993. Nuclear techniques in soil science. Trends in Agricultural Sciences: Soil Science, Amsterdam, vol. 1, pp. 27–46.
- Crestana, S., Mascarenhas, S., Pozzi-mucelli, R.S., 1985. Static and dynamic three-dimensional studies of water in soil using computerized tomographic scanning. Soil Science, USA 140, 326–331.
- Crestana, S., Vaz, C.M.P., 1998. Non-invasive instrumentation opportunities for characterizing soil porous systems. Soil Tillage Res. 47, 19–26.
- Fattah, H.A., Upadhyaya, S.K., 1996. Effect of soil crust and soil compaction on infiltration in a Yolo loam soil. Trans. ASAE 39, 79–84.

- Kutílek, M., Nielsen, D.R., 1994. Soil Hydrology. Cremlingen-Destedt: Catena Verlag, 370 pp.
- Macedo, A., Crestana, S., 1998. Microtomografia de raios X como uma nova ferramenta para estudos em solos. Revista Brasileira de Ciência do Solo, Campinas (in Portuguese, with English abstract), submitted for publication.
- Macedo, A., Cruvinel, P.E., Inamasu, R.Y., Jorge, L.A.C., Naime, J.M., Torre-Neto, A., Vaz, C.M.P., Crestana, S., 1998. X-ray microtomography dedicated to agriculture. Computer and Electronics in Agriculture, Amsterdam, submitted for publication.
- Moore, D.C., Singer, M.J., 1990. Crust formation effects on soil erosion processes. Soil Sci. Soc. Am. J. 54, 1117–1123.
- Morin, J., Van Winkel, J., 1996. Effect of raindrop impact and sheet erosion on infiltration rate and crust formation. Soil Sci. Soc. Am. J. 60, 1127–1223.
- Posadas, D.A.N., Tannús, A., Panepucci, H., Crestana., 1996. Magnetic resonance imaging as a non-invasive technique for investigating 3D preferential flow occurring within stratified soil sample. Computers and Electronics in Agriculture, vol. 14, Amsterdam, pp. 255–267.
- Rawls, W.J., Brakensiek, D.L., Simanton, J.R., Kohl, K.D., 1990. Development of a crust factor for a Green–Ampt model. Trans. ASAE 33, 1224–1228.
- Rosenfeld, A., Kak, A.C., Reconstruction. In: Rosenfeld, A., KaK, A.C., Digital Picture Processing. 2nd ed., vol. 1, Chapter 8, Comput. Sci. Appl. Math. Academic Press, San Diego, 1982, pp. 353–430.
- Slattery, M.C., Bryan, R.B., 1994. Surface seal development under simulated rainfall on an actively eroding surface. Catena supplement 22, Catena Verlag, Cremlingen-Destedt, pp. 17–34.