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Original Research

Influence of Building Material Rubble and Thermal Stress on the Structural Properties of Soil – Mercury Porosimetry Studies

Grzegorz Bowanko*

Institute of Agrophysic PAS, Doświadczalna 4, 20-290 Lublin, Poland

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Abstract

This study was conducted to analyze the effects of the presence of rubble on soil porosity. Brick, concrete, and aerated autoclaved concrete, 10% by weight, were added to soil samples. The changes in soil properties were analyzed in the course of thermal stress – alternating the temperature of the soil samples between -20ºC and +30ºC. Humidity was kept at a constant 25%. The study was conducted on two groups of samples. Both groups consisted of soil mixed with building materials and one group contained an additional 6% by weight of peat as a reclamation factor. The measurement of the pore size distribution was performed on a mercury porosimetr AUTOPORE IV 9500, of MICROMRTRICS. In the course of the study it was demonstrated that the presence of building materials had a significant impact on soil porosity, and that the extent of this impact varied throughout the course of thermal stress.

Keywords: mercury porosimetry, urban soil, building materials, thermal stress

Introduction

As a result of human activity, the area of anthropogenic soils is increasing from year to year, and the rise in urban soil represents a significant percentage of these soils. The greatest degradation of the soil environment occurs in urban areas and these areas are characterized by destruction of the natural soil profile [1, 2]. Large areas of cities are covered with various types of buildings and these can restrict or prevent the normal circulation of air and water. Rubble, in various forms, is left in the soil during the course of construction work and these contaminants corrode due to the impact of weather and the soil environment and, in turn, impact upon the properties of the soil itself [3-5].

Porosity is one of the characteristic qualities of soil that is changed when building materials are added to the soil. The number of pores in any soil, their volume and size, influences important phenomena such as the retention and circulation of gas, water, and nutrients, as well as soil penetration by roots [6-8].

Under prevailing field conditions, however, the large inter-aggregate pore spaces are dewatered (rapidly) and then most of the water and nutrients fluxes occur in smaller intra-aggregate pores [9, 10] with the limited inter-aggregate fluxes through the contact areas between aggregates, [6]. In the inner and usually wetter part of aggregates, impaired oxygen supply may affect biological activity [11] and mineralization of plant nutrients, and increase the losses of nitrogen by denitrification [7]. Aggregate formation and the associated rearrangement of particles also results in an increased apparent thermal diffusivity as compared with homogenized material [11]. Soil surface aggregates and associated pore structure have the potential to greatly influence water vapor adsorption and evaporation [12, 13]. In sealing soils, detailed characterization of PSD is helpful in the description and modeling of soil and crust transport properties [14].

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*e-mail: gbowanko@ipan.lublin.pl

For this reason, the porosity and pore distribution should be taken into account when considering the structure of soil [15, 16]. The porosity of the soil is determined by the granulometric composition, degree of coating of grains, types and amounts of minerals, and the amount of humus, plus climatic conditions (temperature, humidity), vegetation, tillage use and fertilization, and drainage [17].

While there are many studies into the challenges posed by urban soils, the influences of the presence and corrosion of building materials within these soils is largely ignored. The aim of this study was to analyze the changes in porosity of urban soils as they are subjected to thermal cycles (all samples having been contaminated by building materials and half the samples having been additionally modified by the inclusion of peat).

Materials and Methods

The study was conducted on loessial soil samples (Typic Entrochrept wg FAO) taken from the top layer of the profile (0-15 cm) and sifted through a 1 mm sieve. Building materials such as bricks, concrete, and aerated autoclaved concrete (AAC) were then used to prepare the samples. Selected materials are typical construction materials manufactured in accordance with European standards: brick (PN-EN 771-1:2011), aerated autoclaved concrete (PN-EN 771-4:2004; this research used AAC type 600 – d= 600 kg/m^3), and concrete (PN-EN 206-1:2003)

Selected building materials were obtained by smashing them with a hammer and grinding in a ball mill and then sifting through a 1 mm sieve. The soil also was passed through the sieve. Before mixing, all samples were dried at room temperature. The prepared samples of soil and building materials were mixed at a weight ratio of 9 parts soil to 1 part of either brick, concrete, or AAC, and were stored at room temperature. Each sample weighed 100 g (dry weight) and was kept in a plastic bottle. Two identical groups of samples were prepared. Peat (Eutric Histosol) was added (as a reclamation factor) to one group of samples. This peat was dried at room temperature, sifted through the sieve, and added in the ratio 6% by weight. For each thermal cycle the same set of samples was prepared.

In order to standardize the samples, all were moistened with water (25%w.) to a level between the field water capacity and wilting point for loessial soil. They were then subjected to several cycles of drying-wetting to standardize the aggregate composition.

The samples were then subjected to cyclic temperature changes from -20ºC to 30ºC, at a constant humidity of 25%. They were first frozen at -20ºC and stored at this temperature for one week. After this they were heated and stored at 30ºC for one week. The following week the samples were frozen and the next week samples were heated, etc. This cycle was maintained for 18 weeks. First samples for analysis were taken after 6 weeks (6 week – first cycle). The rest of the study material was frozen and heated in the next weeks. Second samples for analysis were taken after the next 6 weeks (12 week – second cycle). The rest of the study material was frozen and heated in the next weeks. The last samples were taken after the next 6 weeks (18 week – third cycle).

Granulometic analysis was performed using the aerometric method of Bouyoucos modified by Cassagrande and Pruszyński. For the latter analysis the soil samples were dispersed using a 0.5% Calgon (sodium metahexaphosphate) water solution. Specific surface area of the samples was measured based on nitrogen adsorption. Specific surface area was measured on QUADRASORB SI (Quantachrome Instruments). Soil samples were dried at 105ºC before analysis. Then samples were put into an analytical burette and part of the burette bulb was placed in the furnace (105ºC). After pre-treatment the burette with the studied material was transferred to a Devar reservoir with liquid nitrogen and then the analysis started.

Content of organic carbon in samples was measured using a TOC MULTI N/C 2000, HT 1300 carbon/nitrogen analyzer (Analytik Jena). Samples are incinerated in a furnace at 1,300ºC.

To determine the pore size distribution, an AUTOPORE IV 9500 (MICROMRTRICS) mercury porosimeter. was used. Before each measurement, the samples were heated to 105ºC in order to remove water retained in the soil aggregates, and were then degassed up to a vaccum that achieved 6.67 Pa pressure at 20ºC.

In order to determine pore radius (r), depending on the pressure of mercury (p_m), it was assumed that these values would accord with the Washburn equation:

$$p_m = 2\sigma_m cos\alpha_m/r$$

...where: σ_m is the surface tension of mercury, and α_m is the angle of wetting of the material by mercury (assumed for all samples to be equal to 140).

Pore size distribution in a function of its radius, dV/dlog(r), was determined assuming that the pores are cylindrical. Due to the fact that the pore radius range included several orders of magnitude, the distribution function was calculated using the logarithm of the radius:

$$\mathrm{d}V/\mathrm{dlog}(r) = 1/v_t\,[v(r_{i+1}) - v(r_i)]\,/\,[\log(r_i) - \log(r_{i+1})]$$

...where: $v(r_i)$ is the volume of the pores having a radius less or equal to r_i, v_t and pore volume is measured at a maximum pressure of mercury: $v_t = v(r_{min})$.

The average size of the radius of the pores in the size range tested $r_{śr}$, calculated from the formula:

$$r_{śr} = 1/(2v_t)\,\Sigma\,(r_i + r_{i+1})\,(v_{i+1} - v_i)$$

The mercury porosimetry method is able to determine the pore distribution of a limited size range (10-0.001μm). The results of these measurements are presented in graphic form:

1) relationship total cumulative volume (TCV) of the logarithm of the radius,

Table 1. Characteristics of soil material.

Probe	Granulomertic composition (%)			S_N ($m^2 \cdot g^{-1}$)	C_{ORG} (%)	pH (in H_2O)
	Sand	Dust	Clay			
Probe without peat						
Soil-s	5	59	36	19.06	1.3	7.93
s-brick	27	40	33	16.34	1.41	8.22
s-concrete	45	34	21	19.63	1.34	11.58
s-foam concr	42	40	18	37.99	1.55	8.09
Probe with peat						
Soil-s	29	41	30	17.55	4.39	6.27
s-brick	41	34	25	13.5	4.09	6.18
s-concrete	40	38	22	13.39	3.68	9.76
s-foam concr	47	31	22	30.1	4.45	7.52

Sand – 2.0-0.05 (mm), Dust – 0.05-0.002 (mm), Clay – <0.002 (mm); S_N – specific surface area, C_{ORG} – organic carbon.

Table 2. Characteristics of soil material after 18 weeks of thermal cycles.

Probe	Granulomrtic composition (%)			S_{N2} ($m^2 \cdot g^{-1}$)	C_{ORG} (%)	pH (in H_2O)
	Sand	Dust	Clay			
Probe without peat						
Soil-s	32	37	31	21.93	1.28	8.35
s-brick	25	53	22	17.68	1.09	8.37
s-concrete	47	38	15	20.7	1.12	8.91
s-foam concr	49	37	14	44.15	1.22	8.25
Probe with peat						
Soil-s	21	57	22	11.59	3.54	7.1
s-brick	48	34	18	10.78	3.35	7.33
s-concrete	41	42	17	9.44	3	7.59
s-foam concr	50	35	15	18.56	3.32	7.92

Sand – 2.0-0.05 (mm), Dust – 0.05-0.002 (mm), Clay – <0.002 (mm); S_N – specific surface area, C_{ORG} – organic carbon.

2) Pore size distribution ($dv/d\log r$) of the logarithm of the radius.

As indicated, samples were removed and tested at 6-week intervals. The results were analyzed for changes in porosity during the thermal cycles. In order to determine the significance of the results an analysis of variance was performed.

Each physicochemical measurement was performed in triplicate. The individual measured values were analyzed for their changes under the influence of thermal cycles performed. In order to determine whether these changes are not due to measurement error, and are the result of changes occurring in the soil material, variance analysis was performed. Statistical study was conducted at a significance level $\alpha=0.05$. Statistical tests were performed using Microsoft Excel 2010.

Results and Discussion

Basic characteristics of the soil material used in this study are shown in Table 1. The granulometric composition of the soil indicates that it can be classified as a heavy textured soil (36% clay). According to Feret's Triangle, the soil also can be classified as a silty soil common. Soil mixtures used in this study, in terms of the granulometric composition, can also be classified as medium-textured soil (mixtures with brick and concrete) and light-textured soil (mixtures with AAC). Depending on its composition, the specific surface area of the analyzed mixtures is smaller than the natural soil (mixtures with brick) or bigger (mixtures with concrete and with AAC). Organic carbon content is higher in the mixtures used than in the natural soil. Values of pH of the samples after the addition of building materials

Table 3. Porosity characteristics of the soil and its mixtures before and after the cyclical changes of temperature.

Model urban soil	Before cycle changes of temperature				After cycle changes of temperature			
	Total cumulative volume ($mm^3 \cdot g^{-1}$)	Total porosity (%)	Bulk density ($g \cdot cm^{-3}$)	Average pore radius (µm)	Total cumulative volume ($mm^3 \cdot g^{-1}$)	Total porosity (%)	Bulk density ($g \cdot cm^{-3}$)	Average pore radius (µm)
Probe without peat								
soil	101.10	18.60	1.70	1.25	152.63	28.38	1.80	2.97
soil-brick	133.17	23.43	1.68	1.58	116.97	24.15	1.74	1.97
soil-concrete	121.46	21.98	1.84	2.48	142.81	24.85	1.74	3.07
soil-AAC	232.52	33.92	1.36	1.98	235.63	36.21	1.49	0.07
Probe with peat								
soil	159.79	28.28	1.77	1.24	130.12	23.29	1.79	1.98
soil-brick	155.61	26.45	1.70	1.58	98.25	17.59	1.79	1.24
soil-concrete	130.71	21.03	1.90	1.57	109.45	21.02	1.92	2.48
soil-AAC	291.56	39.65	1.36	1.98	244.14	33.20	1.36	0.09

Values in each column within each aggregate size and depth followed by different letters are significantly different ($P < 0.05$).

increased in all cases. After the addition of the concrete, the difference between the natural soil and its mixture was greater than four units of pH.

The addition of peat into the second group of samples resulted in a decrease in the content of clay and reduced the size of their specific surface area. Unsurprisingly, the addition of peat to the samples significantly increased the content of organic matter by several percentage points and decreased the pH level in all samples.

Significant changes in the characteristics of the samples, in all measured parameters after the full 18 weeks of thermal cycles, are shown in Table 2.

After heating cycles were performed, the most significant changes in glanulometric composition were observed in the natural soil, where there was an almost equal distribution of pore sizes. However, in the mixed samples, the thermal cycles did not produce any significant change. In all mixed samples, the content of clay decreased in favour of the content of dust. Changes in temperature caused an increase in the specific surface area of the natural soil and of the mixed samples without peat. The average amount of change exceeded 10% and the largest changes were observed in the natural soil (15%) and in soil mixed with AAC (16.2%). In contrast, the specific surface area decreased in those samples containing 6% of peat, and the average change was over 30%. The greatest reduction in specific surface area was observed in soil mixtures with AAC with peat (54%) and soil with peat (34%). The thermal cycles produced some changes that were matched throughout all the samples. In every case there was a reduction in the levels of organic carbon. The pH level also reduced in all samples and lead to a near equalization of pH levels between the natural soil and the mixed samples.

Porosimetry results of research in Table 3 and in Figs. 1-4 are the average of 5 measurements taken. Table 3 shows the porosimetric measurements performed before and after the thermal cycles.

The porosity of the soil used in the experiment was changed after both thermal cycles and when added to the peat. Thermal cycles caused an increase in the porosity of the soil. Total cumulative volume (TCV) and total porosity (TP) increased by more than half. Pore size changed the most after thermal cycles increased the average pore size (doubled). The additional of peat increased soil porosity, too, whereas average pore radius decreased slightly. Soil with peat after thermal cycles reduced its porosity.

The addition of building materials has increased porosity of mixtures compared to the natural soil. The biggest changes were found in the porosity of the soil in the mixture with AAC. Thermal cycles also changed the level of porosity of mixtures. All mixtures increased their TP. Soil mixture with concrete and AAC increased the TCV, and a mixture of the soil with brick slightly decreased TCV. After conducting thermal cycles, soil mixture with concrete and brick were less porous than natural soil. Whereas a mixture of soil with AAC (though minor changes) were still significantly more porous than natural soil after the same thermal stress.

After the addition of peat to the mixtures, porosity of all samples increased. Carried out thermal cycles on mixtures have reduced their porosity. Most decreased porosity of soil mixtures with brick.

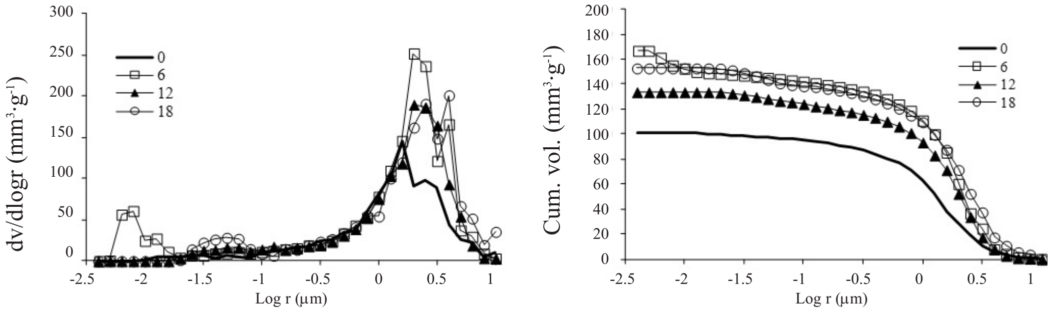
In all tested samples, except a mixture of soil with AAC (and also peat), the thermal cycles caused an increase in the average size of the pore radius.

Statistical evaluation of porosity variability in the samples, in the course of thermal cycles, are shown in Table 4.

Designated coefficients of variation are significant, suggesting a significant effect of thermal cycles on the porosity of the samples tested. The biggest variability of the results were found in the average pore size, especially in the case of mixture soil with AAC.

Changes in the porosity of the samples were the result of changes in pore size, disintegration, and the creation of new aggregates [6, 7, 11]. Samples containing 6% of peat had initially increased in porosity but after the thermal cycles were performed, a marked reduction in porosity was observed [18]. The changes in porosity of these samples were the result of self-generated soil-forming processes that occurred due to the presence of constant humidity and the additional sources of organic matter [7]. These conditions promote the formation of new structures that are characterized by a lower porosity than that of those samples that were not modified by the inclusion of peat [19].

Without peat



With peat

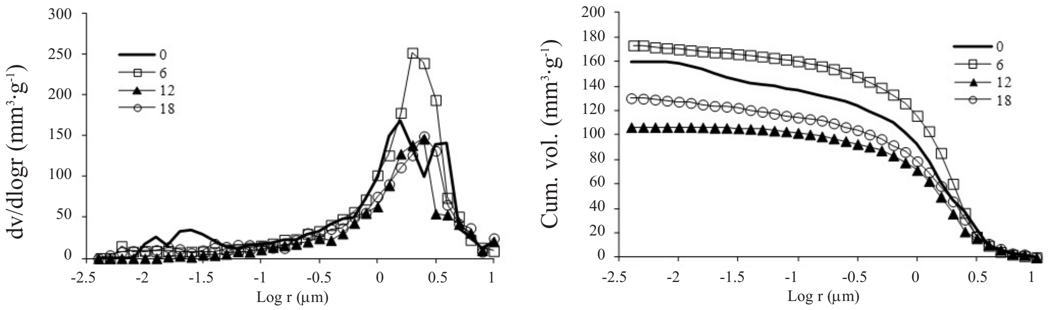
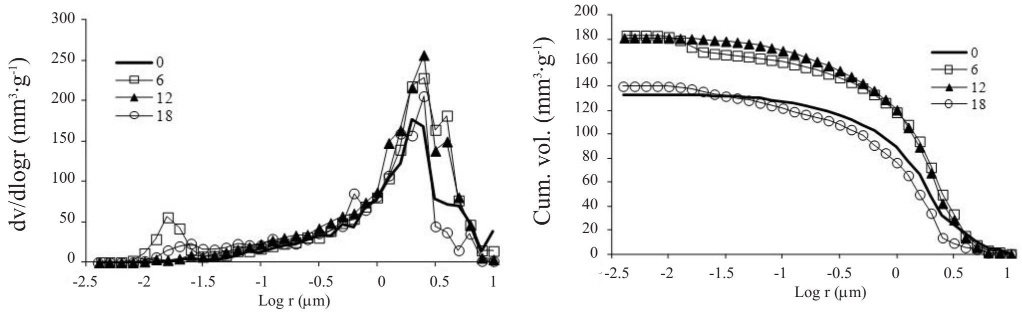


Fig. 1. Changing the porosity of the soil.

Without peat



With peat

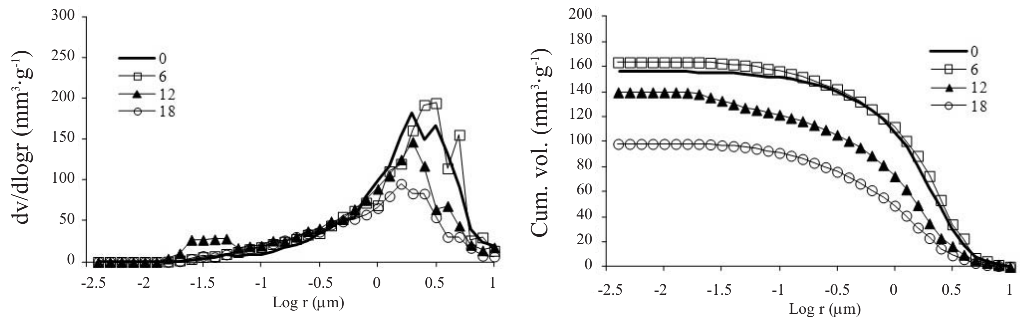
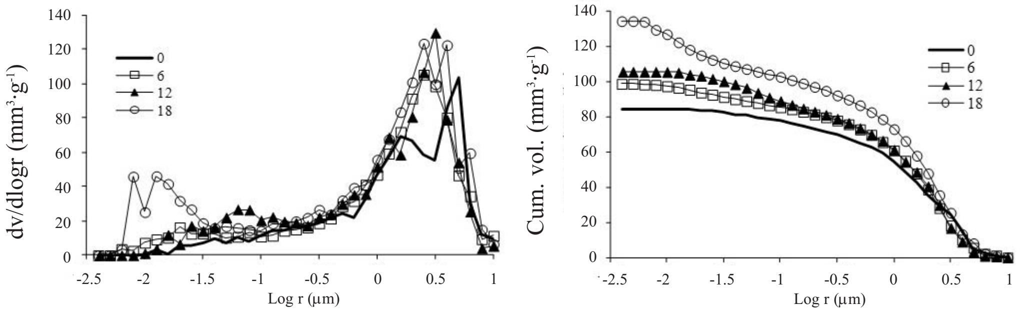


Fig. 2. Changing the porosity of the soil mixture with brick.

Figs. 1-4 show the changes in the porosity of the samples throughout the course of the thermal cycles. In the graphs, 0 indicates the initial porosity of the samples, 6 indicates the measurements after 6 weeks of thermal cycling, 12 being the results after 12 weeks, and 18 being the results after 18 weeks.

The figures show the individual curves selected from the three measurements taken after each thermal cycle (for single samples). The differences between individual curves were not significant. In the samples the biggest changes in total cumulative volume followed in the range of pore sizes in the range 3.16 to 0.79 μm. These changes are confirmed in the pore distribution dv/dlogr graph. Only in the case of soil mixture AAC increase did pore size have two maxima. First in the range of pores of 2.0 to 0.13 μm and the second in the range of 0.1-0.02 μm. The ranges of changes in

Without peat



With peat

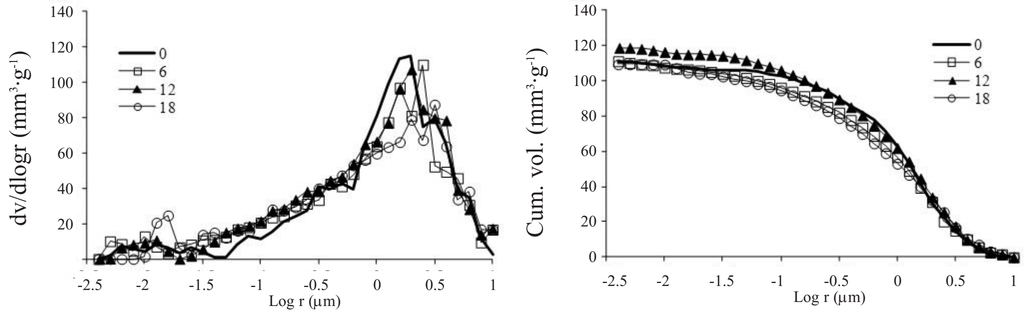
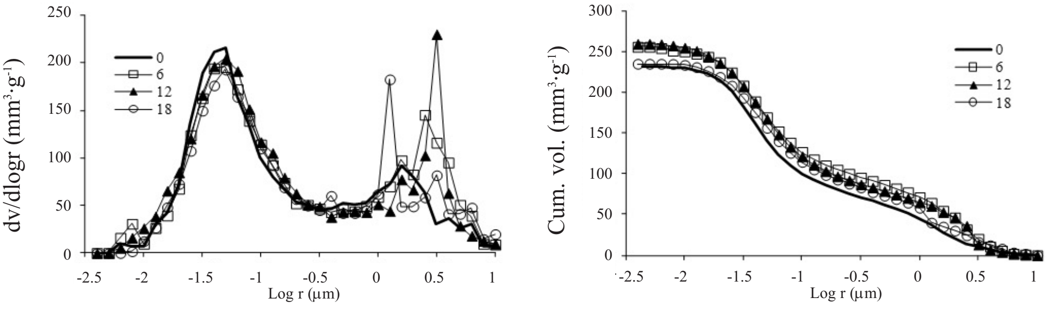


Fig. 3. Changing the porosity of the soil mixture with concrete.

Without peat



With peat

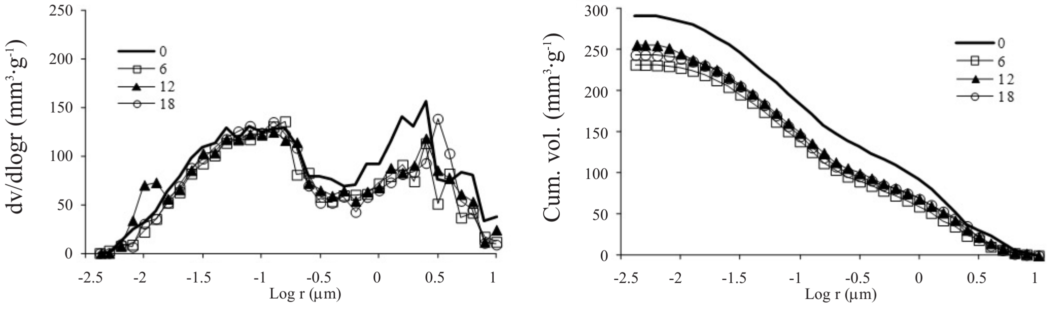


Fig. 4. Changing the porosity of the soil mixture with AAC.

Table 4. Statistical evaluation of variability in soil porosity its mixtures obtained in the course of thermal cycles.

	Total Cumulative Volume				Average Pore Radius				Total Porosity			
Probe without peat												
	6	12	18	fact.	6	12	18	fact.	6	12	18	fact.
soil	+	+	+	20.4	+	+	+	44	+	+	+	21.4
soil-brick	+	+	+	16.4	+	-	-	10.6	+	+	+	14.4
soil-concrete	+	+	+	19.7	+	+	+	27.9	+	+	+	15.9
soil-AAC	+	+	+	5.7	+	+	+	188.7	+	+	+	4.9
Probe with peat												
	6	12	18	fact.	6	12	18	fact.	6	12	18	fact.
soil	+	+	+	21.1	+	+	-	20.8	+	+	+	21.1
soil-brick	+	+	+	20.9	+	+	+	30.9	+	+	+	18.7
soil-concrete	+	+	+	3.8	+	+	+	22.5	+	+	+	1.7
soil-AAC	+	+	+	10	+	+	-	162.5	+	+	+	9.6

(+) – significant change from a statistical point of view, (-) – change irrelevant from a statistical point of view, (fact.) – coefficient of variation, allows comparison of sets of results, expressed in %.

porosity in the samples without peat and with peat are similar. Only the magnitude of these changes are smaller in samples with peat.

According to Figs. 1-4 (the sample of natural soil without peat), the biggest increases in porosity occurred after the first thermal cycle (6 weeks). The low temperature in combination with the presence of capillary water causes crushing of soil material by disintegration of large aggregates nascent ice [20]. Analysis of pore size distribution showed that the change in the porosity of natural soil was shifted toward large pore range. Porosity measured after subsequent thermal cycles did not exhibit significant changes in the size of pores, but there was an increase in their number. In the case of soil mixtures with building materials without peat, the largest changes in porosity occurred after the first cycle (6 weeks) and there was an increase in number of pores without a significant shift of the range of pore sizes.

Analysis of the changes in pore volume of the samples without peat, taken after thermal cycling was performed, again showed that there were major changes in pore volume, especially in the large pores range. Subsequent thermal cycles led to only minor changes in each group and, depending on the composition of the sample, resulted in an increase of porosity (natural soil or its mixture with concrete) or reduction of porosity (mixture with brick or AAC).

Adding peat to the soil and its mixtures caused increased porosity of the starting sample. Thermal cycles also affected the level of sample porosity. The biggest change in the porosity of the soil and its mixtures was found in the largest (2-3 microns) and smallest (0.01-0.1 microns) pores.

According to Figs. 1-4, the thermal cycles of all the soil samples with added peat led to a decrease in porosity. In the natural soil sample there was, after the first thermal cycle, a major increase in porosity across the entire range of pores but subsequent thermal cycles led to a significant decrease in porosity. After the first thermal cycle, the sample of soil with brick produced little or no change in its porosity, but the later cycles led to a substantial reduction of porosity. The soil samples with concrete and the inclusion of peat were the least sensitive to changes in temperature. The changes in the porosity of the subsequent cycles were not significant, and fluctuated around baseline values.

The examination of the peat-modified soil samples that were mixed with AAC revealed that, after the first thermal cycle, there was a decrease in porosity. The presence of peat caused significant changes in the characterization of porosity in the entire range of pores. The most significant changes were seen in the smallest pores. Adding peat to the mixture reduced by half the number of small pores. In pore size distribution there was a significant shift in pore sizes from small and large pores to medium ones when compared with samples without peat. Subsequent thermal cycles slightly increased pore volume over the entire range.

Changes in pore structure due to composition of the samples and thermal stress may influence aggregate stability. Earlier studies revealed that aggregate mechanical strength generally increases with decreasing porosity and thereby a greater number of contact points between soil particles appear [21, 22] that are influenced by the balance between changes in macro- and microporosity [14]. Interrelation of the pore size ranges can contribute to greater aggregate strength from the denser subsoil than the topsoil as shown in earlier studies [23].

In addition, the results of this research showed that mercury intrusion porosimetry is a useful tool for the quantification of soil degraded by the rubble effects on changes in aggregate pore size distribution in a wide range of pore radii. Usefulness of the method also was shown while characterizing pore size distribution in response to soil tillage systems [24], land use types [7], saturation, and drying [25, 26]. Thus, our results and those from the literature results support the acceptance of mercury intrusion porosimetry as a standard method to characterize pores of solid materials [27] and the International Union of Pure and Applied Chemistry (IUPAC) [28, 29]. However, we should be aware that mercury intrusion porosimetry does not actually measure the internal pore size, but rather determines the largest connection from the sample surface toward that pore [30] and simplifies all pores as a bundle of capillary pores and therefore does not detect pore shape and tortuosity. In addition, MIP does not consider clay swelling so that the pore size distribution may be different from that determined by water saturation [26]. By using the non-swelling loess soil in the present study we could avoid possible effects of the swelling phenomena on pore size distribution. An earlier study [7] on similar soil found that the concentration of pore volume of pore radius 7.5 to 0.1 μm obtained from the water retention curve was greater than from the mercury intrusion porosimetry method. The above differences can be attributed to the different measurement procedures that include dewatering of pores under suction while determining the water retention curve and intruding mercury under pressure in the mercury intrusion porosimetry.

Conclusions

The above results indicate that the porosity of both the natural soil and mixed samples is closely related to the composition of the sample and the duration of the thermal cycles. The addition of rubble of building materials in the soil increased the porosity of samples compared to the natural soil. The thermal cycles led to significant changes in the structure of the prepared urban soil. These changes affected both total pore volume and pore size distribution. Thermal cycles resulted in an increase of the number of small and large pores, and the greater increment was observed in the number of large pores.

The soil mixed with building materials became less sensitive to changes in their structure due to thermal cycles, which appears to be a positive outcome. The addition of peat in the early stages of the experiment increased the porosity of all the samples. However, the thermal cycles resulted in a reduction in the porosity of all samples. The presence of peat led to a reduction in growth of small pores as a result of the thermal cycles.

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