Influence of Micro and Nanosilica on the Frost–Heave Process

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Abstract. Frost-heave of soils is an important problem in engineering practice. Thus, it became necessary to find an adequate method that would reduce the negative effect of this phenomenon on building structures. The paper presents the preliminary tests results of microsilica (MS) and nanosilica (NS) influence on the water freezing process in frost-susceptible soils. The selection of stabilising additives also allowed for the analysis of changes that result from their transition from micro to nanoscale. For the purposes of the experiment a test stand was constructed, that enables to analyse six samples at the same time, at inflow of water from the bottom and freezing direction from the top. The use of linear potentiometer displacement sensors allowed to measure the increase in the height of samples automatically. Additionally, the measurement stand was equipped with digital semiconductor temperature sensors, so temperature could be measured at various heights of the samples. Moreover, an array of electrodes was applied to measure the electrical conductivity of soil at various levels of samples, which enabled the analysis of changes that occur in freezing soil. The tests were conducted according to the BS 812-124:1989 standard, in three variants: soil, soil + 5% MS, soil + 5% NS. The obtained results demonstrated that the addition of microsilica reduces the occurrence of ice lenses, which in turn significantly decreases the growth in sample height. However, the addition of nanosilica completely stops the frost-heave process. Ice lens is not created, only a frost line is noticeable. Analysing the distribution of temperatures at different heights, it was noted that stabilised samples are characterised by higher temperature in comparison to the soil without additives. This means that the analysed stabilisers reduce the degree of freezing of the soil mixture. The highest temperatures were noted in samples stabilised with nanosilica. Conductivity tests also demonstrated that the smallest changes occurred in samples stabilised with nanosilica. This results from the fact that ice lenses did not occur in such samples. In order to analyse the obtained results and their interpretation thoroughly, it is necessary to perform a detailed analysis of the microstructure both of soil and of soil with stabilising additives.

1. Introduction

The development of civil engineering makes it more necessary to erect structures on subsoil characterised by complex, disadvantageous conditions, including also frost-susceptible soil.

The frost-heave phenomenon often leads to damage and deformation of the roads structures as well as the building structures. It is thus necessary to find a method to eliminate soil susceptibility to the freezing and thawing processes.

Generally, one of the methods of ground improvement is the application of various stabilising additives. Soil stabilisation is divided into two main categories: mechanical and chemical [1, 2]. The
newest trend in the technology of chemical soil stabilisation is the application of nano-additives. The most commonly used among them is nanosilica [3, 4].

So far, the conducted research on the influence of nanosilica on the formation of the soils geotechnical parameters referred only to studies on the basic physical and mechanical properties [3, 5]. On the other hand, no comprehensive studies that would enable to determine the influence of the nano-additive on the properties and behaviour of frost-susceptible soil are available. Some authors only mention the possibility to apply nanosilica to reduce the increase in volume as a result of freezing [6].

Additionally, there are some research on the positive changes in parameters that have an indirect effect on frost heave (permeability, compactibility) of soils stabilised with microsilica [7]. Thus, one may suppose that with the same stabilising additive but in nano-scale the tendency of changes in soil parameters will be similar. However, they should occur on a much higher scale, which results from the properties of nanomaterials [8].

The aim of this study is to recognise the influence of micro and nanosilica on the course of the water freezing process in frost-susceptible soils, including the size and distribution of ice lenses, temperature distribution and changes in the volume of soil. Moreover, the application of selected stabilising additives allowed for the analysis of changes resulting from the transition from micro to nano scale.

2. Materials
The soil selected for the study was frost-susceptible, which grain size distribution was determined with use of hydrometer analysis according to the PN-EN 1997-2:2007 standard [9]. Based on the grain size distribution curve (figure 1), it was determined that it was sandy clayey silt saclSi [10].

One of the stabilising additive was microsilica (MS), which is commonly used as an additive to concrete. The grain size distribution of the silica fume was determined with use of laser granulometer analysis.

The second stabilising additive was nanosilica (NS) in colloidal form, type Levasil 200/30. The selected product is commonly used in the construction industry. In order to determine the distribution of the size of nano-particles of the analysed colloid, tests were conducted in the Zetasizer Nano particle characterisation system.

Specific surface area and pH tests were also conducted both for the soil and for the additives. The characteristics of soil and of the applied additives are presented in table 1.

| Type of material | Average particle size mm | Specific surface area m²·g⁻¹ | pH  |
|------------------|--------------------------|-----------------------------|-----|
| saclSi           | 0.25                     | 35.70                       | 8.65|
| MS               | 1.80 ·10⁻²               | 68.31                       | 8.75|
| NS               | 9.00 ·10⁻⁶              | 196.49                      | 9.00|

The differences in the grain size distribution of specific additives in comparison to the analysed soil are presented in figure 1.

3. Method
Laboratory tests were performed in three variants: soil without additives, soil with an addition of 5% microsilica and soil with an addition of 5% nanosilica.

The proportions of content of specific additives and the methods of mixing them with soil were determined pursuant to the study by Kalkan [7] for microsilica and to the works by Seyedi, Gelsefidi and Mamaghanian [5] and Bahmani et al. [3] for nanosilica.

The main aim of the tests was to analyse the samples at optimum moisture content w_{opt}, which corresponds to maximum soil dry density ρ_{ds}. 

2
In order to determine the above parameters, tests were conducted in the Proctor automatic compactor, pursuant to the ASTM D698 standard [11]. The obtained curves of compactibility for individual soil mixtures are presented in figure 2.

The tests of the frost-heave phenomenon were conducted according to the BS 812-124:1989 standard [12], in a Weiss C 600 climate chamber adapted for this purpose. Six samples from each variant were tested. Samples with diameter of 100 mm and a height of 110 mm were uniformly compacted to the maximum dry density of soil.

In the climate chamber, soil samples were placed on an automated measurement stand created especially for the purposes of the discussed tests. This stand combines the solutions proposed by Zhou et al. [13] (temperature and displacement sensors) and Kang and Lee [14] (array of electrodes) and it allows for the analysis of 6 samples at the same time. Tests were conducted in an open system with free water inflow. These assumptions reflect the conditions in which ice lenses emerge.

In order to recreate such conditions, an insulated water tank was used, from which the water got into the samples by the capillary rise during the freezing process. In order to maintain the temperature of the
water in the tank above zero, a temperature controlling device was connected, to keep the water temperature at approximately 5°C. Temperature could be controlled with use of sensors placed in the tank.

The structure of the stand ensures constant experimental conditions without interfering with the measurement system. Each sample was equipped with four digital semiconductor temperature sensors, which enabled to analyse temperature changes throughout sample height. Additionally, seven pairs of electrodes were placed in each sample, at different height, enabling to analyse the freezing process by measuring the electrical conductivity of horizontal soil layers (figure 3). Additionally, linear potentiometer displacement sensors allowed for a precise, automated measurement of the increase in sample height (figure 4).

Figure 3. Schematic diagram of soil sample with electrodes and temperature sensors

Figure 4. Schematic diagram of the measurement stand

During the tests, all measurement data were recorded with use of an automated data measurement acquisition system connected to the computer. The whole test stand is shown in figure 5.
4. Results and discussion

The obtained results based on the measurements of 6 samples from each variant (soil, soil + 5% MS, soil + 5% NS) were averaged and presented in form of diagrams.

4.1. Increase in height

Due to the conditions of the experiment, the increase in height resulted from the emergence of ice lenses. The highest increases (by approx. 30%) were noted in samples of pure soil. The application of microsilica reduced the increase in height on average by 20% in comparison to samples of soil without additives. On the other hand, the addition of nanosilica eliminated the increase in height completely: the ice lens did not emerge at all, only the frost line could be observed (figure 6).

![Figure 6. Increase in the height of samples with time](image)

4.2. Temperature

Based on the conducted tests, the distribution of temperature on specific levels, numbered from the bottom of the sample, was determined and presented in graphic form (figure 7).
The results have shown that the temperature distribution varied depending on the applied stabilising additive. In spite of the identical conditions in the climate chamber, it is visible that after 120 hours in the frost zone (levels 1, 2) the lowest temperatures were noted for samples of pure soil, followed by...
samples with the addition of silica fume. For samples stabilised by nanosilica, during the whole process of freezing, the temperature on both levels was higher than 0°C. This may result from the fact that stabilising additives reduce the pores spaces. The smaller the pores, the slower the heat exchange as well as the lower the temperature necessary for the water to freeze inside them [15, 16]. This is why no ice lenses emerged in samples stabilised with nanosilica.

4.3. Conductivity
The conducted tests enable to analyse changes in electrical conductivity during freezing on each of the 8 levels numbered from the top of the sample. The course of changes in electrical conductivity in specific samples during the test is presented in figure 8.

The obtained results clearly illustrate the differences in changes of electrical conductivity in time, depending on the type of stabilising additive. The analysis of changes in conductivity in the frost zone (levels 5, 6, 7) allows to state that the process of freezing in soil without additives took place much faster and that its scope was greater than in samples stabilised with microsilica. For samples with an addition of nanosilica, the conductivity in this zone decreased only slightly, which confirms the lack of emergence of ice lens.
Figure 8. Changes in electrical conductivity in time: a) saclSi, b) saclSi + 5% MS , c) saclSi + 5% NS

5. Conclusions

The conducted tests allowed for a preliminary recognition of the influence of micro and nanosilica on the process of water freezing in soil and enabled to draw the following conclusions:

- It was noted that a 5% addition of MS significantly reduces the increase in sample height. On the other hand, 5% addition of NS led to a complete lack of ice lenses emergence.
- Differences in temperature distribution in the frost zone allowed to note the correlation: the finer the additive, the higher the temperature inside the sample and thus – the lower degree of freezing.
- The analysis of changes in conductivity demonstrated that the process of creating ice lenses in soil without additives took place much faster and that its scope was greater than in samples with MS. On the other hand, in samples stabilised with NS, the changes in conductivity were negligible, which is confirmed by the absence of ice lenses.
- The description of the phenomena that occur inside the analysed samples requires a detailed analysis of the changes in microstructure both of the soil without additives and soil stabilised with use of various additives.

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