A LABORATORY CHARACTERIZATION OF SOILS AND CLAY-BEARING ROCKS USING THE ENSLIN-NEFF WATER-ADSORPTION TEST

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Abstract

The application of the Enslin-Neff water-adsorption test for the determination of soil-index properties has been investigated on commercially available reference minerals and on real soils. The Enslin-Neff test is a simple and reliable laboratory method, which can provide important information about the behavior of soils, clay-bearing rocks and aggregate fines that are in contact with water. Although the test has been frequently used for bentonite testing, practically no results have been published about the physical meaning of the Enslin-Neff water-adsorption values for real soils. The results of this study indicate that the Enslin-Neff water-adsorption test can be used to obtain accurate values of some soil-index properties, such as the liquid limit, the plasticity index, the methylene-blue value, and the soil-water characteristic curve.

Keywords

water adsorption, methylene blue value, soil water characteristic curve, soil suction

1 INTRODUCTION

A test method for determining the water-adsorption capacity of powders was proposed by Enslin in 1933. It was found to be applicable to viscous silk, cellulose, paint, glue, gelatine, adhesives based on starch, soils and some other substances. The method has, however, been improved several times since then [1]. Today, the method is included in the German industrial norm DIN 18 132 [2], and is known as the Enslin-Neff water-adsorption test (w_A). The test is widely used in clay mineralogy and in the production of bentonite for quality control [3]. Although the Enslin-Neff method is simple, quick to perform, inexpensive and can give important information about the nature and the behaviour of geological materials, practically no results have been published about the physical meaning of Enslin-Neff water-adsorption values for real soils and clay-bearing rocks. Available publications, mostly published by German researchers, deal with improvements to the method [4], [5], the reproducibility of the results [1], and the comparability of the results with the Atterberg-limits values [6]. Dieng [6] investigated natural soils, bentonites and kaolinitic clays, and laboratory-prepared mixtures of kaolin and bentonite with limestone, as well as laboratory-prepared mixtures with lime and organic material in order to define the relationship between the water-adsorption value and the Atterberg-limits values (w_L and I_P). Based on the results of his experiments, he defined the relationships between the w_A and w_L and I_P values as follows:

For soils with:

$$w_A + 0.3\, w_{Ai} \le 210\% \qquad (1)$$

$$w_L = 0.61\left(w_A + 0.3\, w_{Ai}\right) \qquad (2)$$

when: $w_A > 40\%$

$$I_P = 0.28\left(w_A + 0.3\, w_{Ai}\right) - 5 \quad \text{kaolin} \qquad (3)$$

$$I_P = 0.51\left(w_A + 0.3\, w_{Ai}\right) - 13 \quad \text{other soils} \qquad (4)$$

For soils with:

$$w_A + 0.3\, w_{Ai} > 210\% \qquad (5)$$

$$w_L = 1.13\left(w_A + 0.3 w_{Ai}\right) - 126 \qquad (6)$$

$$I_P = 1.1\left(w_A + 0.3\, w_{Ai}\right) - 140 \qquad (7)$$

Dieng [6] defined the value of w_{Ai} as a function of water adsorption after 100 minutes (w_{A100}) and water adsorption after 5 minutes (w_{A5}):

$$w_{Ai} = w_{A100} - w_{A5} \qquad (8)$$

Compared with the Atterberg-limit values for soil classification, the advantage of the Enslin-Neff method is that only a very small sample, about 3 grams, is needed for the test. This means that the test could be used to identify the nature of soils, rocks and aggregate fines even in cases when other test methods cannot be used due to the lack of samples of an adequate size, for instance when studying fine-layered or fractured expansive clay-bearing rocks and the slip surfaces of rocks, when testing the nature of fines in the aggregate production industry, for forensic and other purposes.

This paper presents the results of a study in which the results obtained for the Enslin-Neff water adsorption (w_A) were compared with those obtained by a test of the Atterberg limits (w_L, I_P), the methylene-blue test (MB_f), and the soil-suction measurements for determining the soil-water characteristic curve (SWCC) [7].

2 METHODS

Water-adsorption values were determined according to the German norm DIN 18 132 [2] using a conventional Enslin–Neff device (Figure 1). Oven-dried samples with weights of 0.3 grams, in the case of bentonites, and 1 gram in the case of other powders, were placed on the frit using the recommended funnel method. For each material in the experiment, three parallel tests were performed in order to check the reproducibility. The readings of the burette were recorded continuously, with the first reading after 15 seconds, and then following the power law. The final reading was taken after 24 hours. The effect of evaporation from the burette was determined prior to the experiment. With the increasing duration of the test the effect of evaporation on the readings increases. For most of the soil samples in the experiment, the water uptake in the sample was finished after 15 minutes. For the reference bentonites the readings lasted for more than 24 hours, so that the error due to evaporation could be relatively large.

For the purpose of this study, the readings after 24 hours were used as a reference for all three bentonites.

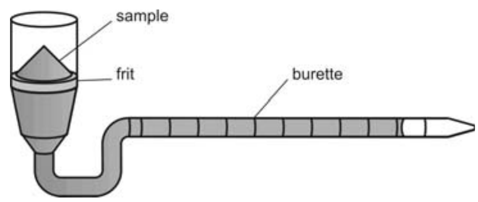


Figure 1. The Enslin-Neff device used in the experiment.

The Atterberg-limits test was determined according to the standard SIST TS CEN ISO/TS 17892-12:2004 [8]. For the determination of the liquid limit a 60g/60° cone was used. The reference samples were used in the natural state, without sieving, whereas in the case of the real soil and clay-bearing rocks, only fines passing through a 0.063-mm sieve were used.

The methylene-blue value, expressed as MB_f, was determined using the standard method defined in SIST EN 933-9:1999 [9]. The qualitative mineralogical composition was determined by X-ray diffraction analysis using a Phillips device with Cu Kα radiation.

The soil suction at low suction rates was determined by the axial-translation technique, using a soil-moisture pressure-plate device. Saturated samples that had been prepared at a water content equivalent to the liquid limit or slightly higher were placed on a porous plate with a high air-entry value, and then maintained at the chosen cell air pressure of 10 kPa, 33 kPa and 100 kPa. At suction rates higher than 100 kPa, a Decagon chilled-mirror dew-point potentiometer was used for the tests.

3 MATERIALS

For the purpose of this study, 134 samples were prepared and tested. The five reference samples consisted of commercially available materials: non-active limestone flour, brick clay and Na^+ and Ca^{2+} bentonites. All the other samples were taken from typical Slovenian soils and clay-bearing rocks, and consisted of mixtures over a wide spectrum of different minerals: Eocene flysch marl and claystone, Oligocene highly overconsolidated grey marine clay and marl, Miocene marl, Pliocene overconsolidated clays from the Sub Pannonian basin, and Pliocene karstic clays (terra rosa). Quaternary alluvial and delluvial soils, taken from weathered zones in alluvial gravels, carbonatic, ultramafic and metamorphic phyllonites were also included in the test program.

Table 1. Characteristics of the soils used in the research.

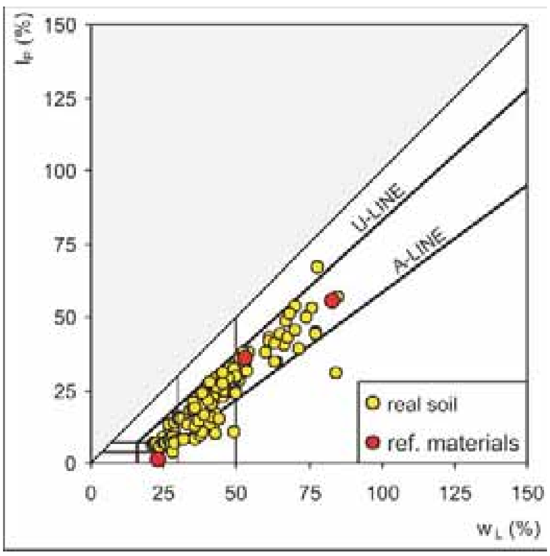
Sample	w_L %	I_P %	w_A %	MB_f g/kg	$w_{s1500kPa}$ %	Dominant mineral*
Bentonite Wyoming	535	490	778	336		M_{Na}
Bentonite Macedonia	207	172	297	176	46	M_{Ca}
Bentonite Slovenia	83	56	113	172		M_{Ca}
Brick clay Slovenia	53	36	73	57	14	I/C
Stone flour Slovenia	23	-	32	2	1	Ca, $CaCO_3$: 99 %
Oligocene marine clay	41-48	26-29	62-70	48-25	11-12	MM:I/K/Ca/Q/
Eocene flysch	42-50	25-32	56-64	56-60	11–13	MM: I/K/Calc/Q/
Miocene marl	39-53	15-34	56-74	20-31	7-11	MM: I/K/M/Calc/Q/
Pliocene clay	52-85	25-57	67-88	61-62	11-23	MM: I/K/M/Q/mica
Pliocene karstic clay	63-122	41-89	83-121	8-13	20-35	L/Mn/Ca/Q
Alluvial clay	67-68	49-51	78-82	38-40	14-15	MM: I/K/M/Q
W – gravel river alluvium	37-42	8-16	47-55	3-12	3–7	MM
W - dolomite	21-38	6-20	29- 51	2–34	2-11	MM: D/I/C
W – phyllonite	27-40	8-19	41-57	7-11	3-9	MM:Sc/C/B/Q
W – serp. harzburgite	40-78	19-42	68-116	28	13-16	S/C/L/O

* M_{Na} – Na montmorillonite > 70 %; M_{Ca} – Ca montmorillonite > 70 %, I- illite/muscovite, B – biotite, C – chlorite, Ca – calcite, D – dolomite, L – limonite, MM – wide mineral mixture, Mn – manganese oxide, O – olivine, Q – quartz, S – serpentinite, Sc – sericite, W – weathered products inside the bedrock

4 RESULTS AND DISCUSSION

The results for the Atterberg-limit values, the Enslin-Neff water absorption, the methylene blue, the water content at a soil suction equal to 1500 kPa and the prevailing mineral composition are given in the ranges for the representative samples of the typical groups of soils (Table 1).

The liquid limits and the plasticity indexes of the tested soils are presented for all the samples in Figures 2 and 3. The reference samples (commercially available materials) are indicated in the key in each diagram. The reference clayey samples were found to be located close to the same straight line, which corresponds to the U-line in the AC diagram, whereas most of the tested real soils



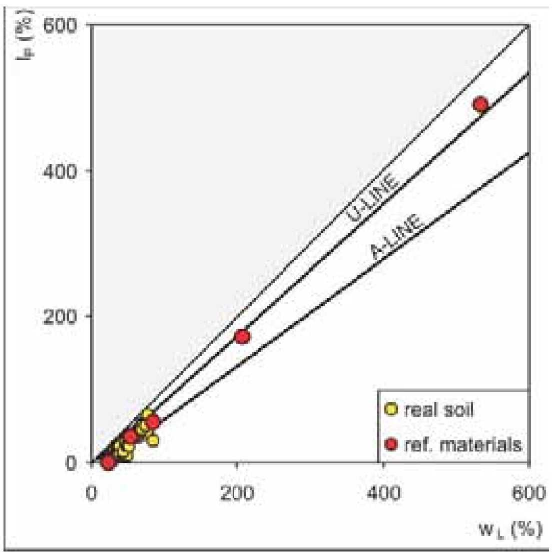


Figure 2. Position of the tested soils in the AC diagram, limited scale (left) and complete scale (right).

lie above the A-line (Figure 2). The U- and A-line are defined as in the ASTM standard D 2487-00 [10], where the U-line is defined as the empirically determined approximate "upper limit" for the natural soil and the A-line as the border line between silts and clays.

4.1 RELATIONSHIP BETWEEN THE ENSLIN-NEFF WATER-ADSORPTION TEST AND THE ATTERBERG-LIMITS TEST

The Atterberg-limits test is one of the most widely used tests in soil mechanics for classification purposes, although it is partially subjective and requires experienced laboratory staff. At least 200 g of soil is needed for the test, which is quite inconvenient, especially when thin layers of highly expansive clay are encountered inside the bedrock mass or when coarse material with a low fines content has to be tested. However, the Enslin-Neff test is a promising test that could overcome the shortcomings of Atterberg's soil-classification test.

The experimentally determined relationship between the Atterberg liquid-limit test and the Enslin-Neff water-adsorption test is given in Figure 3.

For the reference materials an excellent correlation was found between w_A and w_L and the corresponding simple equations can be written as:

$$w_L = 0.69\, w_A \qquad (9)$$

$$w_A = 1.45\, w_L \qquad (10)$$

For the real soils, the values of the coefficient in equation (10) can vary between 1.13 and 1.77. The experimentally defined relationship is close to that proposed by Dieng [6], but it is expressed in a much simpler form.

For the real soils the scattering of the results seems to increase with the increasing liquid limit (Figure 3). Therefore, the coefficient w_A/w_L was calculated for all the samples and the standard deviation was determined to be 0.2. Figure 4 presents the distribution of the coefficient w_A/w_L and the normal distribution for the determined standard deviation and coefficient w_A/w_L. The liquid limit, w_L, could be calculated with a 10% accuracy from the real measurement by using equation (10) for more than 50% of the samples (for example, w_L=40% in the range 36–44%).

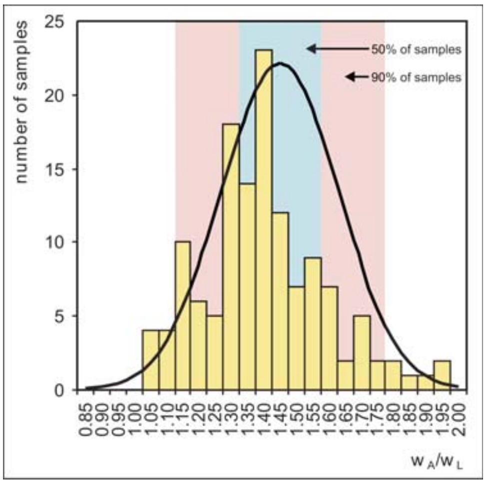


Figure 4. Grouping of the soils following the position in the AC diagram.

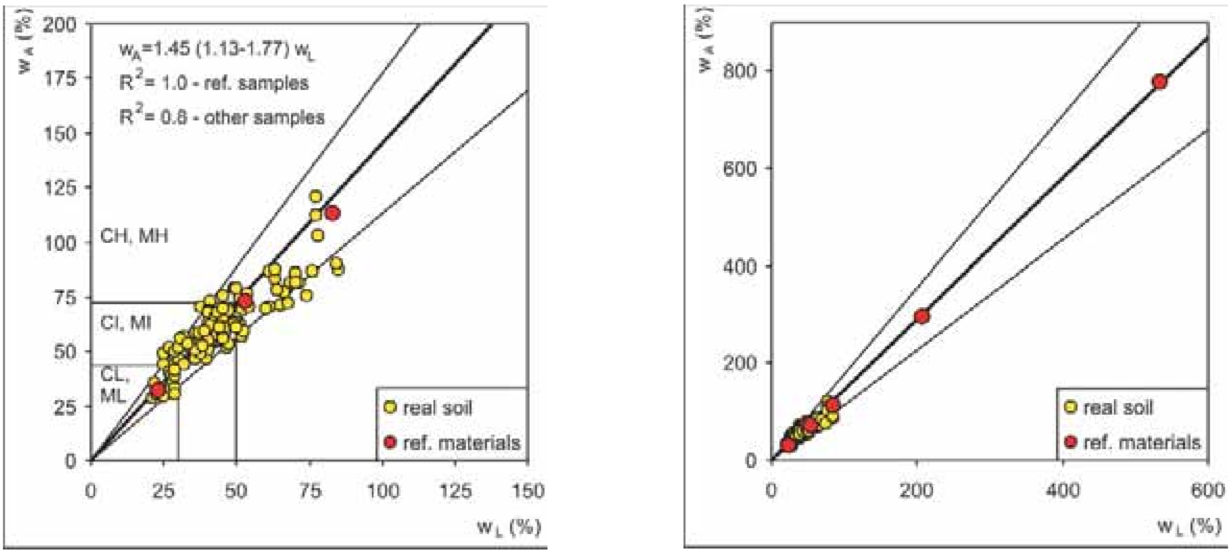


Figure 3. Enslin-Neff water adsorption as a function of the liquid limit, limited scale (left) and complete scale (right).

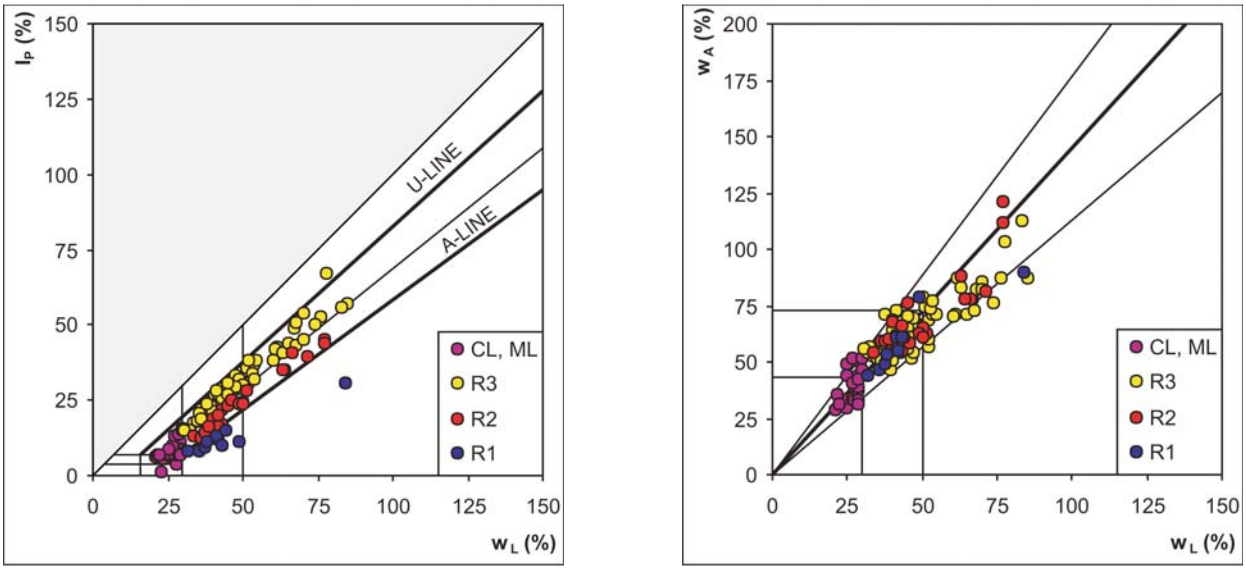


Figure 5. Grouping of soils following the position in the AC diagram (left) and the relationship w_L–w_A (right).

To explain the influence of the different types of soils on the ratio w_A/w_L, the samples were divided into four groups: lean clays and silts represent a special group with a liquid limit w_L < 30%, whereas the groups R1, R2 and R3 were defined according to the soil position in the AC classification diagram (Figure 5).

From Figure 5 it can be concluded that the type of soil does not influence the relationship w_A/w_L and that equation (10) is valid for all types of soils. However, for the relationship between w_A and I_P (Figure 6) these findings are not valid.

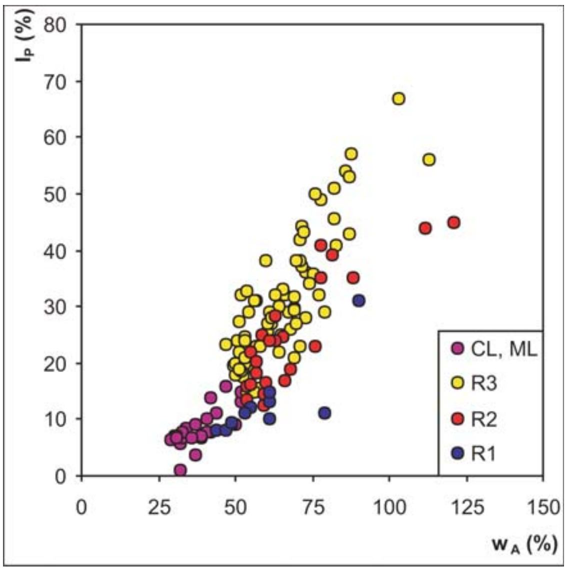


Figure 6. The index of plasticity as a function of water adsorption and soil group.

The results of the experiments show that both values, i.e., w_A and w_L, have similar physical meanings when describing the clean mono-mineral soils as well as the real heterogeneous soils. In the case of both tests, the measured water content belongs to the water adsorbed to the external and internal surfaces of the grains. However, the water content at the liquid limit corresponds to an undrained shear strength of c_u = 1.6 kPa [11], but does not correspond to the maximum void ratio (e_{max}). The sample on the frit in the Enslin-Neff device is in the loosest state, and is completely saturated when the test is finished. The water content obtained from the Enslin-Neff test is therefore higher than the water content at the liquid limit for the specific soil.

The results of the experiments also confirmed that the Enslin-Neff method could replace or provide an excellent complement to the liquid-limit test, especially in cases when limited quantities of samples are available for the test, or when it is difficult to perform the liquid-limit test, as in the case of Na^+ bentonites or low plastic soils and micas.

4.2 RELATIONSHIP BETWEEN THE ENSLIN-NEFF WATER-ADSORPTION TEST AND THE METHYLENE-BLUE VALUE

It is well known that the specific surface area (SSA) and the cation-exchange capacity (CEC) influence the behaviour of clay in contact with water and water solutions. For more than 40 years the methylene-blue (MB) test has been recognized as a rapid and easily applicable test for

SSA and CEC determination [12], [13], [14], [15], and different classification charts for predicting the swelling potential of soils are based on MB values [13], [15], [16]. In its aqueous state methylene blue is a cationic dye, $C_{16}H_{18}N_3S^+$, which can adsorb onto negatively charged surfaces. The MB molecule has a rectangular shape with the dimensions 17 Å*7.6 Å*3.25 Å, and it is assumed that the MB molecule lies on its largest surface [15]. No special equipment is needed for the test. The MB value is determined by measuring the quantity of methylene-blue dye necessary to cover the total (external and internal) surface area of the particles contained in the soil.

From this point of understanding, the Enslin-Neff and the MB tests exploit similar phenomena – the adsorption of water molecules or positively charged dye cations onto negatively charged external and internal particle surfaces. It can therefore be assumed that the correlation between the MB and Enslin-Neff must exist in soils that contain negatively charged surfaces, and where the amount of adsorbing water prevails over the amount of capillary water.

The experimentally determined relationship between the MB_f and w_A values is given in Figure 7. It can be seen that there is no unique and linear relationship between the two measured parameters, which is probably due to the influence of the capillary water and of the different sizes of the adsorbing molecules. The concentration of the results at lower values of MB_f (MB_f < 15g/kg) belongs to the silty soils (group R1), where most of the water in the soil belongs to the capillary water and the particles can be estimated as inert. At values of MB_f > 15g/kg the correlation between MB_f and w_A can be expressed as a potential function with a correlation coefficient of R^2 = 0.54. The correlation coefficient was only calculated for the real soils. The reference bentonites were not included, since the small number of these samples with positions far away from the centre of the group would have drastically improved the correlation coefficient R^2.

However, in the literature it has been reported that the MB shows a high dependency of the adsorption on the type of cations in/on the clay. Researchers have reported that the adsorption of the MB dye is only complete when the sample is in the lithium and sodium exchanged form [15]. From this point of view it is too early to recognize the Enslin-Neff test as a method that could successfully replace the MB test, but it can certainly complement it. The results of the research are promising and show that these investigations should be continued, taking into account the influence of the specific cation-exchange capacity and the specific surface area of the tested soils on both of the measured parameters.

4.3 ENSLIN-NEFF WATER ADSORPTION – SWCC

The soil-water characteristic curve (SWCC) is a continuous sigmoidal function, represe nting the relationship between the water content (gravimetric or volumetric) or the saturation degree and the stress state of the pore water. Many soil properties can be related to SWCC, such as the volume change, the hydraulic conductivity and the shear strength. The SWCC has three stages that describe the process of desaturation of a soil: the capil-

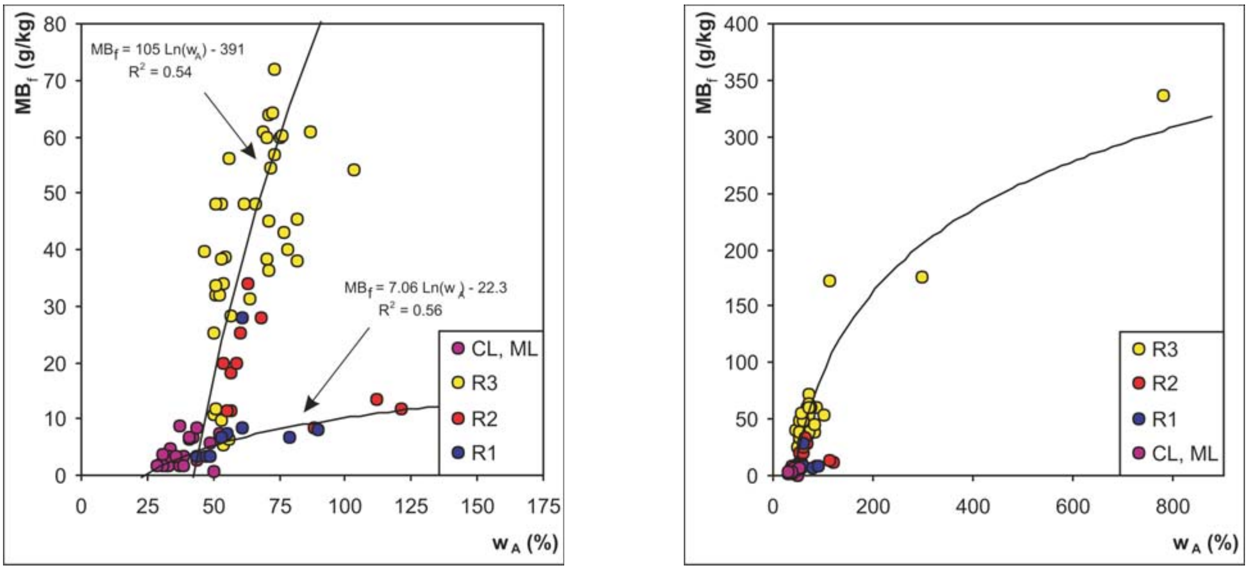


Figure 7. The MB_f – w_A relationship, limited scale (left), complete scale (right).

lary saturation zone, where the pore water is in tension but the soil remains saturated due to capillary forces; the desaturation zone, where the water is displaced by air within the pores; and the residual saturation zone, where the water is tightly adsorbed onto the soil particles and flow only occurs in the form of vapour [7].

The capillary saturation zone ends at the air-entry value, where the applied suction overcomes the capillary water forces in the soil and air enters the pores. The amount of water in the soil or the water content at a certain suction stress depends on the soil's density and the soil's stress history. However, each type of soil has a characteristic maximum void ratio in its loosest state (e_{max}), at which it can hold the highest amount of water in the saturated state (at zero suction). This point corresponds to the first point on the SWCC, which can be called, following the similarity with the saturated soil oedometric compression curve, the "virgin" SWCC.

The laboratory tests for SWCC determination at low suction usually start on samples that have been prepared at the liquid limit, which means that the soil is no longer in the loosest state. Following the description of Dieng [6], the Enslin-Neff water adsorption can correspond to the first point on the "virgin" SWCC at which the soil pores and the diffusive double layers are filled with water. A relationship between the water content at zero suction and the water content at the residual water content must therefore be expected.

Figure 8 shows the SWCC curves of four representative soils used in the investigation. For each soil sample the experimentally determined soil suctions are given for

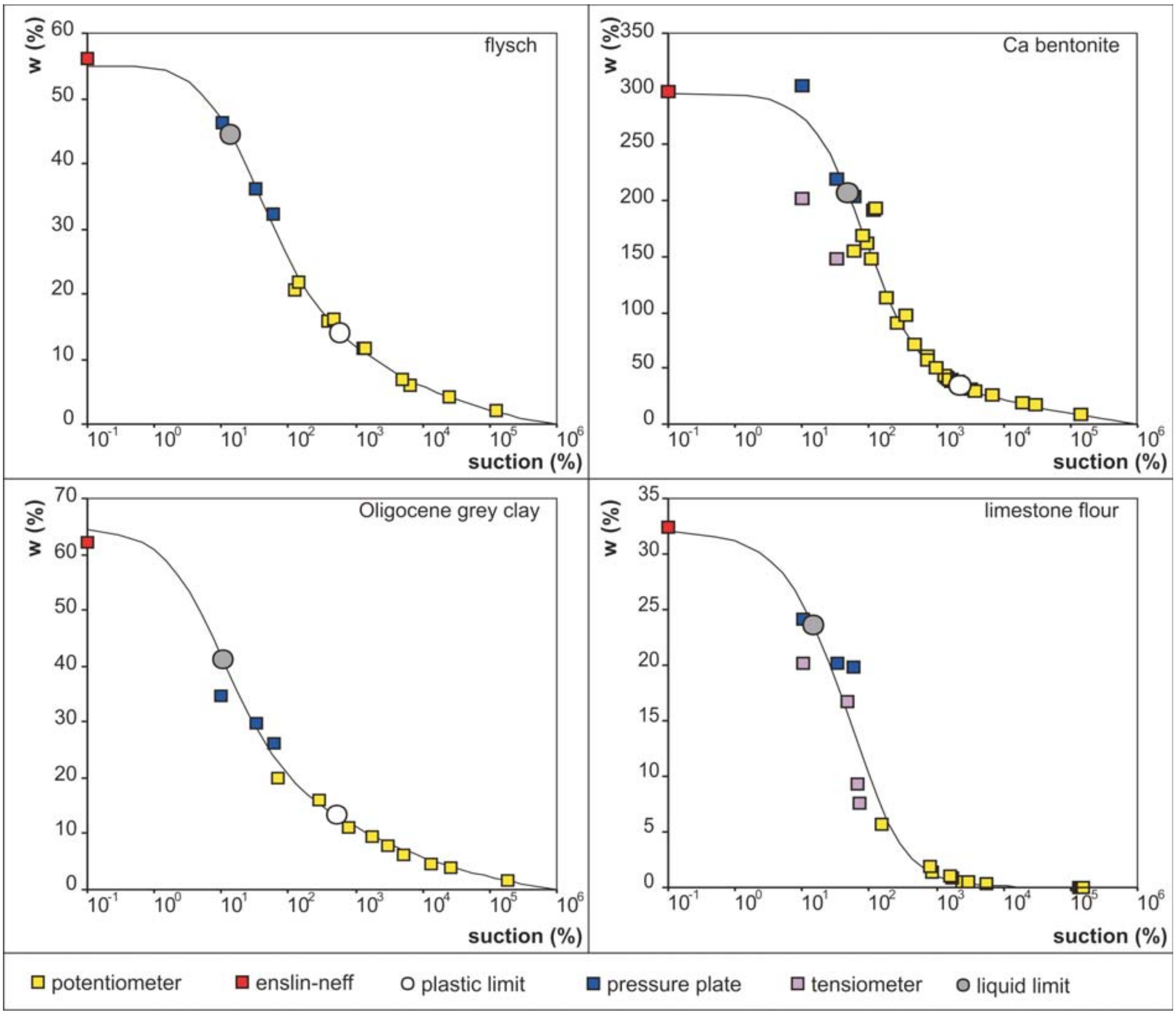


Figure 8. SWCC curves for four representative soils with the positions of the liquid limit and the plastic limit.

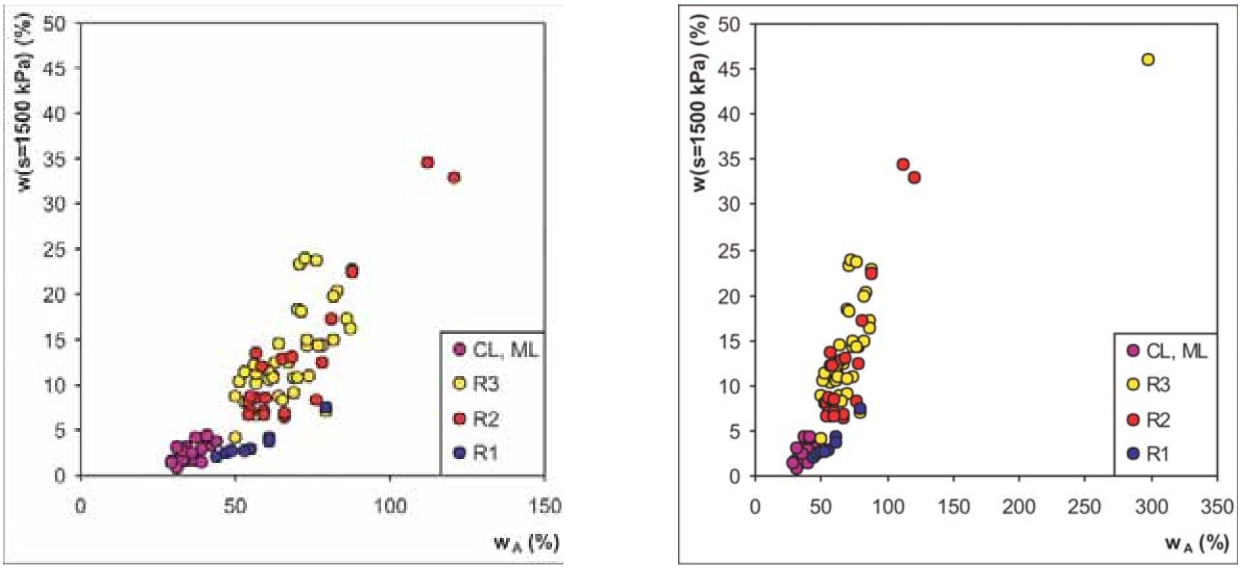


Figure 9. Water content at a suction of 1500 kPa, as a function of w_A.

certain water contents, and the values of w_A, w_L and w_P are indicated in the graph. The SWCC is described using the Fredlund–Xing equation [17]. Different researchers have investigated suction at the liquid and plastic limits of different soils and have found different corresponding values between 0.5 and 30 kPa [18], [19], [20].

The typical results presented in Figure 8 show that the magnitude of the suction at the liquid limit of each specific soil depends on its Enslin-Neff water adsorption. This means that the value w_A, which corresponds to the maximum water content at zero suction, also controls the suction at the liquid limit.

The relationship between w_A and the water content at a suction of 1500 kPa is given in Figure 9. At least two different types of behaviour can be observed. The first type belongs to the soil group R1, for which the w_A values are lower than 80%, and it corresponds to a water content at a suction of 1500 kPa of less than 7.5%. The second type belongs to the soils with w_A values higher than 50% and a water content at a suction of 1500 kPa of more than 7.5%. The amount of experimental data is too small to describe the relationship between the two parameters, but the Enslin-Neff water adsorption could be estimated as the first point on the virgin SWCC.

5 SUMMARY AND CONCLUSION

An experimental program was conducted in the laboratory with the aim of examining the physical meaning of Enslin-Neff water adsorption (w_A) in terms of some of the more widely known index properties of soils: the Atterberg limits, the MB_f value and the SWCC. Real soil samples with different origins and mineralogy were used, together with clean, commercially available limestone flour, brick clay and bentonites, to determine whether the results are comparable for different adsorption ranges. The following conclusions may be drawn from the investigation:

- Linear trends are evident in the relationship between the Atterberg limits and the Enslin-Neff water adsorption. This observation confirmed the results published by Dieng [6]. By introducing a simplified equation (9), the classification of cohesive soil or fines can be reasonably predicted from the Enslin-Neff test. The Enslin-Neff test is faster, requires a very small sample, and is less subjective than the Atterberg-limits test.
- Trends are indicated in the relationship between the MB_f and the Enslin-Neff above values of $w_A > 60\%$. The strong influence of the nature of the exchangeable cations on the MB_f needs further investigation in the direction of the MB_f and the w_A relationship. However, the obtained results are promising, and could be complemented by further investigations.
- The w_A was recognized as being probably the first point on the "virgin" SWCC. Special connections were recognized between the w_A and the water content at the permanent wilting point. From this point of view, the Enslin-Neff test provides new possibilities for its use in the field of swelling-soil classification methodology after additional testing and certain modifications.

The experimental results have confirmed the wide possibilities for the extended use of the Enslin-Neff method in the laboratory for soil and rock mechanics and for the testing of aggregates. It is especially suitable for soil classification and the preliminary estimation of the possible volume changes of soils and clay-bearing rocks. As it is less subjective than the Atterberg-limits test and the MB titration method, it is a pity that the method has remained almost forgotten and was not introduced to soil mechanics laboratories earlier.

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