

# Soils and Rocks

An International Journal of Geotechnical  
and Geoenvironmental Engineering



Volume 35, N. 3  
September-December 2012

# ***SOILS and ROCKS***

An International Journal of Geotechnical and Geoenvironmental Engineering

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*Soils and Rocks* publishes papers in English in the broad fields of Geotechnical Engineering, Engineering Geology and Geo-environmental Engineering. The Journal is published in April, August and December. Subscription price is US\$ 90.00 per year. The journal, with the name “Solos e Rochas”, was first published in 1978 by the Graduate School of Engineering, Federal University of Rio de Janeiro (COPPE-UFRJ). In 1980 it became the official magazine of the Brazilian Association for Soil Mechanics and Geotechnical Engineering (ABMS), acquiring the national character that had been the intention of its founders. In 1986 it also became the official Journal of the Brazilian Association for Engineering Geology and the Environment (ABGE) and in 1999 became the Latin American Geotechnical Journal, following the support of Latin-American representatives gathered for the Pan-American Conference of Guadalajara (1996). In 2007 the journal acquired the status of an international journal under the name of Soils and Rocks, published by the Brazilian Association for Soil Mechanics and Geotechnical Engineering (ABMS), Brazilian Association for Engineering Geology and the Environment (ABGE) and Portuguese Geotechnical Society (SPG). In 2010, ABGE decided to publish its own journal and left the partnership.

### ***Soils and Rocks***

|              |                       |
|--------------|-----------------------|
| 1978,        | 1 (1, 2)              |
| 1979,        | 1 (3), 2 (1,2)        |
| 1980-1983,   | 3-6 (1, 2, 3)         |
| 1984,        | 7 (single number)     |
| 1985-1987,   | 8-10 (1, 2, 3)        |
| 1988-1990,   | 11-13 (single number) |
| 1991-1992,   | 14-15 (1, 2)          |
| 1993,        | 16 (1, 2, 3, 4)       |
| 1994-2010,   | 17-33 (1, 2, 3)       |
| 2011,        | 34 (1, 2, 3, 4)       |
| <b>2012,</b> | <b>35 (1, 2, 3)</b>   |

ISSN 1980-9743

CDU 624.131.1

**SOILS and ROCKS**

An International Journal of Geotechnical and Geoenvironmental Engineering

**Publication of****ABMS - Brazilian Association for Soil Mechanics and Geotechnical Engineering****SPG - Portuguese Geotechnical Society****Volume 35, N. 3, September-December 2012****Table of Contents****ARTICLES**

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## ***Articles***

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# Correlating Soil Porosity and Respective Geological Unit in Paraíba do Sul Valley, Brazil - A Geostatistical Methodology Proposal

P.I.M. Camarinha, S.J.C. Simões, G.P. Bernardes, I.C.B. Trannin

**Abstract.** This manuscript aims proposing a methodology for correlating soil porosity to the respective geological units using geostatistical analysis techniques, including interpolation data by kriging. The site studied was in Lorena municipality, Paraíba do Sul Valley, southeastern Brazil. Specifically all studies were carried out within an area of 12 km<sup>2</sup> located at Santa Edwiges farm. The database comprehended 41 soil samples taken at different geological and geomorphologic units at three different depths: surface, 50 cm and 100 cm depth. The geostatistical analyses results were correlated to a geological mapping specifically elaborated for the site. This mapping accounts for two different geological formations and a geological contact characterized by a shearing zone. The results indicate the existence of a significant relationship between the soil porosity and the respective geological units. The studies revealed that the residual soils from weathered granitic rocks tend to have higher porosities than the residual soils from weathered biotite gneiss rocks, while the soil porosity within the shearing zone is relatively un-sensitive to the respective geological formation. The spatial patterns observed were efficient to evaluate the relationship between the soil porosity, geology unit and the and geomorphology showing a good potential for correlating with others soil properties such as hydraulic conductivity, soil water retention curves and erosion potentials.

**Keywords:** geostatistic, sampling, error prediction, porosity map.

## 1. Introduction

The knowledge of the physical properties of the soil contributes to improve our understanding of their mechanical and hydraulic properties. However, depending on the size of the area of interest and the proposed objectives, awareness of these physical properties, in plot field, can be very expensive and time consuming. In different research domains such as agronomy, mining, hydrology and river basin planning, where large portion of land has to be analyzed, the plot field rarely contributes to satisfactory results, requiring assess to spatial variability of soil properties. In geotechnical engineering, mapping surveys of areas susceptible to mass movement have provided to be an important tool to reduce - or avoid - the potential losses from natural hazards, besides to providing important information for decision-makers regarding the land use occupation. Therefore, spatial information can provide a wider view of the environment studies and allow correlation with other properties or attributes related to physical landscape elements.

A practical limitation found in studies of soil properties is that, generally, the amount of the good-quality field

datasets are scarce for a particular area of study. In the same way, when one wants to integrate data from other landscape elements for example, a geological map, the difficulty becomes even greater in areas of small dimensions, due to the lack of thematic maps produced in appropriate scale.

For these reasons, it is important to develop low-costs methodologies that provide tools for spatial representation of a certain property. Among the available options, the spatial data interpolation methods are commonly used to meet these needs. They are capable of predicting the spatialization of a random variable for large or small areas based on punctual observations (sampled points).

Burrough (1998) states that, when there are enough data, most interpolation methods produce similar values. However, in the case of sparse data, such methods have limitations in the representation of spatial variability, since they do not consider the location of the samples and then ignore the continuity of the phenomenon. Krige (1951) initiated studies seeking to understand the spatial variability of concentration of gold, considering the spatial location of each sample and its interference in neighboring occurrences.

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Submitted on February 16, 2011; Final Acceptance on November 21, 2012; Discussion open until April 30, 2013.

Based on studies of Krige (1951), Matheron (1963, 1971) proposed the theory of geostatistics, known as the *Theory of Regionalized Variables*, which gives the theoretical basis for the application of kriging. According to Vieira (2000), kriging has the ability to produce better estimates among the interpolation methods because it is based on two premises: non-bias estimator and minimum variance estimates.

Several studies were then developed using kriging in order to describe the spatial distribution of soil properties. Vieira *et al.* (1981), for example, investigated the spatial dependence of the infiltration rate of water on Yolo (CA) clay loam. In their study, the spatial variability of 1280 field-measured infiltration rate was studied using geostatistical concepts. Depending on the correct selection of samples, it was observed that a minimum of 128 samples was enough to obtain nearly the same information as with 1280 samples.

Gomes *et al.* (2007) made use of kriging techniques to know the spatial distribution of soil density, concentration of organic matter and soil texture in order to find relations with land use occupation. The study was carried out with 165 point samples distributed over an area of 47 km<sup>2</sup>, located at Ribeirão Marcela Basin, MG, Brazil. The authors found that some types of agricultural soil use causes direct interferences in the spatial distribution of soil attributes. Thus, they emphasized the importance of the use of kriging to find critical areas regarding the soil management and, also, to provide important information for proper planning of the land use.

Fernandes da Silva *et al.* (2007) analyzed some geotechnical properties such as grain size distribution fractions and plasticity index, in Ubatuba area, north coast of São Paulo State, Brazil. The purpose of the study was to find spatial patterns of these properties that allow the estimation of geotechnical behavior of soils. The authors employed three interpolation methods (*Nearest-neighbor interpolation*, *Weighted mean* and *Kriging*) using 73 point samples in order to find the better technique to estimate the spatialization of the geotechnical properties. The results suggested that kriging is a better model to be used in regionalization of the parameters.

From the previous studies presented, kriging technique has proved to be an important tool for spatial representation of soil properties. However, kriging and any other spatial interpolation method aggregate errors in their estimates, which is often overlooked in geostatistical studies. In some cases, the error associated with interpolation method can be so high that causes large discrepancies between estimates and observed reality.

It is important to know the error associated with the prediction in order to evaluate the results obtained. Besides, to validate and to refine the methodology applied it is also important to seek additional spatial information regarding the investigated area. This can be done, for example, corre-

lating the spatial of a certain soil property with other field spatial information previously known, such as geology, geomorphology and pedology.

Based on the fundamentals proposed from Matheron (1963, 1971), this study investigate the existence of spatial dependence of the variable porosity ( $\eta$ ) for an area of 12 km<sup>2</sup> in Lorena municipality, located in the Paraíba do Sul Basin, São Paulo State. These analyses were done for three different depths: surface (samples taken between 0 and 20 cm); 50 cm and 100 cm respectively. The study presents a methodology for correlating soil porosity to the respective geological unit using geostatistical analysis techniques, including interpolation data by kriging and the error involved.

## 2. Materials and Methods

### 2.1. Study area

The study area comprises the region of Santa Edwiges Farm, which is inserted in the region upper Taboão stream watershed, located in the Paraíba do Sul Valley, Southeast of Brazil (Fig. 1).

Despite its small size (12 km<sup>2</sup>), this investigated area was chosen because it reflects the diversity found in geology and geomorphology in the region, which characterizes the transition between the extensive plains of the Paraíba do Sul Valley and the coastal mountain chain of Serra do Mar.

Therefore, the Santa Edwiges farm is entirely inserted into a geological context formed by crystalline rocks of pre-Cambrian age (>500 million years). The map of Fig. 2 shows the various geological units encountered within the study area: a) metamorphic rocks (schists, gneisses and migmatites) of the Embu Complex (Hasui & Sadowski, 1976; Carneiro *et al.*, 1978; Landim, 1984); b) igneous rocks (in most cases are of granitic composition) of the Quebra-Cangalha Suite (Landim, 1984); c) high deformation bands (milonites rocks); d) unconsolidated sediments located in the floodplain of small streams.



**Figure 1** - Location of study area.

Based on Fig. 2 one can see that the northern portion of the area consists of the *Embu Complex*. This unit consists of metamorphic rocks (gneisses) having in its composition minerals more easily weathered such as mica and feldspar. Thus, the soils from these rocks are usually fine-grained soils where predominate clay minerals favoring the development of a more impervious and more homogeneous soil. The soils are usually thicker and have a reddish color because of the presence of iron-rich minerals such as biotite.

The unit *Granitoid Quebra-Cangalha* occurs in the southern area. It is composed predominantly of white to grey leucocratic granites. Rocks of this unit are composed of minerals more resistant to alteration such as quartz and feldspar. Due to the presence of these minerals, soils are predominantly whitish, have a sandy-clay constitution and with a significant presence of mineral fractions of coarser material, such as silt. The coarser texture of these soils and the absence of vegetation, favors the occurrence of erosion processes in advanced stages, such as ridges and ravines.

Mylonite rocks account for about 10% of the total area studied. These areas were subjected to intense tectonic tensions in ductile conditions, that is, at depths greater than 10 km (Ramsay, 1980). For this reason, they have a well developed foliation and the presence of well-structured and fine-grained minerals like mica and chlorite as a result of processes of retro metamorphism due to percolation of fluids in shear zones. While on the surface, the intense foliation of these rocks facilitates the processes of weathering and the formation of soils with a high proportion of clay.

The unit *Fluvial Terrace* and the unit *Unconsolidated Sediments* are associated with a fluvial plain Ribeirão Taboão and its main tributaries. In this area are identified paleo-terraces with pelitic sediment composition (silt and clay) and, secondarily, sand and angular pebbles of quartz and feldspar. Dark-colored sediments are also observed indicating the presence of rich organic soil.

Regionally, this area is inserted in the geomorphological unit of Mid Plateau of the Valley of *Paraíba do Sul*

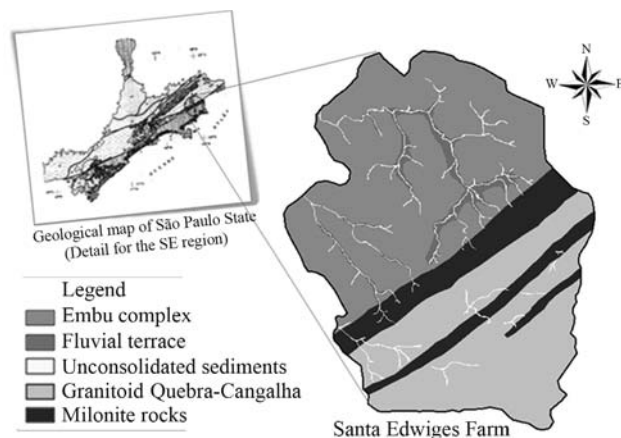
which was described by Ponçano *et al.* (1981) in the Geomorphological Map of the State of São Paulo. The diversity of geological substrate as described above is directly responsible for the wide variation of reliefs and soils found in the Santa Edwiges farm.

On the scale adopted (1:10.000), it was possible to identify, on the basis of morphometric elements of terrain (hypsothetic and slope) three distinct geomorphological units: Ridge Escarpments, Mountain with Moderate to Gentle Hillslopes and Smooth Convex Surface. These units can be observed through a digital elevation model (DEM), developed by Lima (2005) (Fig. 3). The description of these units is as follows.

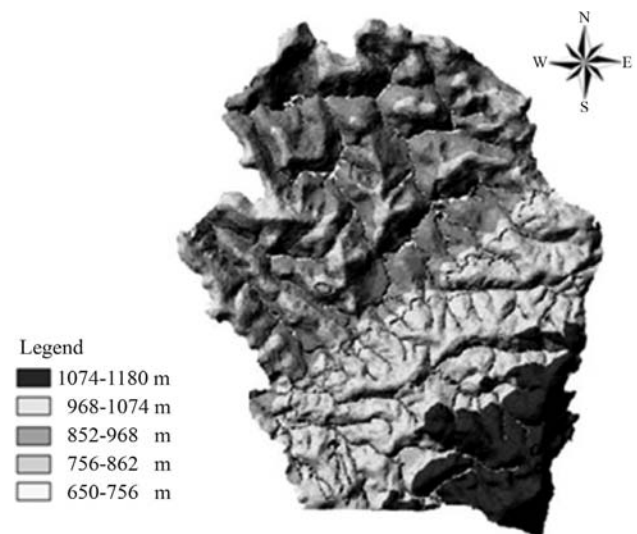
Ridge Escarpments - located in the northern portion of the study area is characterized by relief with steep slopes (>30%) and large amplitudes of altitude (>300 m). Characteristics of this unit include deep narrow valleys and high drainage density. The soils associated with Ridge Escarpments are, predominantly, young residual soil and saprolite.

Mountain with moderate to gentle hillslopes - occupies the central and northern area of study. They feature rounded shapes with medium slopes (>15%) lower than elongated ridge escarpments and amplitudes of altitude ranging between 100 and 300 m. The valleys are more extensive with the presence of alveoli and medium drainage density. The profiles of weathering in this region vary widely, with sites presenting large thicknesses of transported soil.

Smooth Convex Surface - represents the transition between both units above-mentioned situated in the central portion of the study area. It corresponds the areas of very



**Figure 2** - Geological Map of Fazenda Santa Edwiges, drawn to scale 1:10.000, adapted from Rodrigues & Milanezi (2005).



**Figure 3** - Digital Elevation Model for Fazenda Santa Edwiges identifying the existing landscape units: Ridge Escarpments (black), Mountain with Moderate to Gentle Hillslopes (light gray) and Smooth Convex Surface (dark gray). Adapted from: Lima (2005).

low slope (<5%) with low-altimetric variations (<50 m) usually related to alluvial plains. In this region predominates thicker layers of mature residual soil.

## 2.2. Geostatistics

The geostatistics is able to provide estimates in a context governed by a natural phenomenon with distribution in space. It assumes that the values of variables are auto-correlated spatially Landim *et al.* (2002), , such that samples close together in space are more alike than those that are further apart, and is based on the *Theory of Regionalized Variables*, proposed by Matheron (1963).

Geostatistics uses the variogram as one of its primary tools (sometimes called semivariogram) to measure the spatial variability of a regionalized variable, and provides the input parameters for the spatial interpolation of kriging (Krige, 1951; Webster & Oliver, 1993.)

### 2.2.1. Variograms

The variogram is used to investigate the relationship of the distribution of variable ( $z(x)$ ) in space. This tool is able to measure the degree of spatial dependence between samples over a specific support. The expected squared difference between paired data values  $\{z(x_i)$  and  $z(x + h)\}$  to the lag distance  $h$ , are simply used for its construction assuming stationarity in increments, (Landim, 2006).

To obtain an estimate of the parameters a theoretical semivariogram model is used to define the weights of the kriging function. One can formulate an estimator for the semivariogram which may be calculated thus:

$$2\hat{\gamma}(h) = \frac{1}{|N(h)|} \sum_{i=1}^{N(h)} [z(x_i) - z(x_j)]^2, \quad h \in \mathcal{R}^d \quad (1)$$

where  $Y(x_i)$  represents the value of the data at location  $x_i$ ;  $h$  is the displacement between the data pairs; and  $H(h)$  is the number of such data pairs in the region, which is given by:

$$N(h) \equiv \{(x_i, x_j) : x_i - x_j = h; i, j = 1, \dots, n\} \quad (2)$$

When there is spatial dependence, usually, the closest two measures are more alike than two others that are further apart, allowing  $\gamma(h)$  to increase as the distance  $h$  increases too. However, from a certain distance, it will not find related values with  $z(h)$  because the spatial correlation between the samples ceases to exist (Goovaerts, 1997; Landim & Struraro, 2002; Gumiaux *et al.*, 2003). The semivariogram point where the data present no spatial dependence, maintained around the same semi-variance (y axis) and where it is established a straight line in the graph, called the “sill” (C). The distance from the origin (x and y coordinates equals zero) to the sill, is called the “range” (a), which represents the radius of influence of sampling points on its neighborhood, indicated by the distance at which the variance stabilizes (Fig. 4).

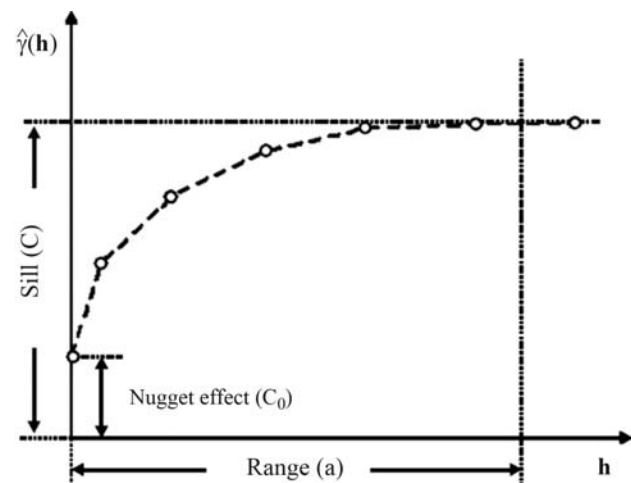
By definition,  $\gamma(0)$  should be zero, but in practice it is noticed that there are cases where as  $h$  approaches zero,  $\gamma(h)$  approaches a positive value called “nugget effect” or “nugget” ( $C_0$ ). This parameter demonstrates the discontinuity of semivariogram for distances smaller than the smallest distance observed among samples. The nugget effect is the value of semi-variance for the distance zero and represents the component of spatial variability that can not be related to a specific cause, that is, random variability (Camargo, 1997), or also to be linked to errors in measurement.

### 2.2.2. Kriging

The kriging method uses information from the theoretical variogram model to find the optimal weights to be associated with points with known values (sampled points) which will estimate the unknown points. In this respect, it is understood as a series of techniques of regression analysis that seeks to minimize the estimated variance from a previous model, which takes into account stochastic dependence among the data distributed in space (Matheron, 1971; Isaaks & Srivastava, 1989).

The difference between kriging and other methods of interpolation is the way the weights are distributed in the different samples. For traditional methods, such as *Simple Linear Interpolation*, all samples have weights equal to  $1/N$  ( $N$  being the total number of samples). In the Inverse Distance Weighting (IDW), the weights given to samples are related to the inverse of the distance that separates the estimated to the observed values. In the case of kriging, it is the weighted mobile average of values observed in the neighborhood where the closest neighbors have more weight and, the neighbors further apart, have increasingly smaller weights, zero and even negative values (Cressie, 1993; Ribeiro Junior, 1995).

Moreover, the kriging provides unbiased estimates and minimum variance. Unbiased estimates indicate that,



**Figure 4** - Parameters of the semivariogram. Adapted from Camargo, 1997.

on average, the difference between the estimated and observed values, through a sampling, for the same point must be zero; and minimum variance means that these estimators have the smallest variance among all unbiased estimators (Camargo, 1997).

### 2.3. Sampling

One of the main questions in studies involving interpolation methods refers to the amount of samples needed to obtain representative results. Generally, the size of the study area is taken as reference. For geotechnical maps in detail scale of 1:10.000, Matula & Pasek (1984-apud Zuquette; Gandolfi, 2004) suggest minimum sampling between 10 to 25. For this scale, Zuquette (1987) recommend 15 sampling points at minimum with a distance between points equal to 258 m. However, some authors, like Webster & Oliver (1993), assert that there is no a specified minimum number of samples for to realize geostatistical studies and emphasize the importance of the findings being complemented by technical knowledge or information of areas similar to the study area.

Due to the wide diversity of landscape of the study region, set in rugged terrain, a specific number of samples were not previously determined. The study sought another path, where the database was developed in several stages to achieve desired quality, guided from observation of the spatial distribution of the variable analyzed and also based on the error attributed to the process. The sampling process also sought to include all geological units in order to search

possible correlations between the pattern of spatial porosity and the Geologic Map shown in Fig. 2.

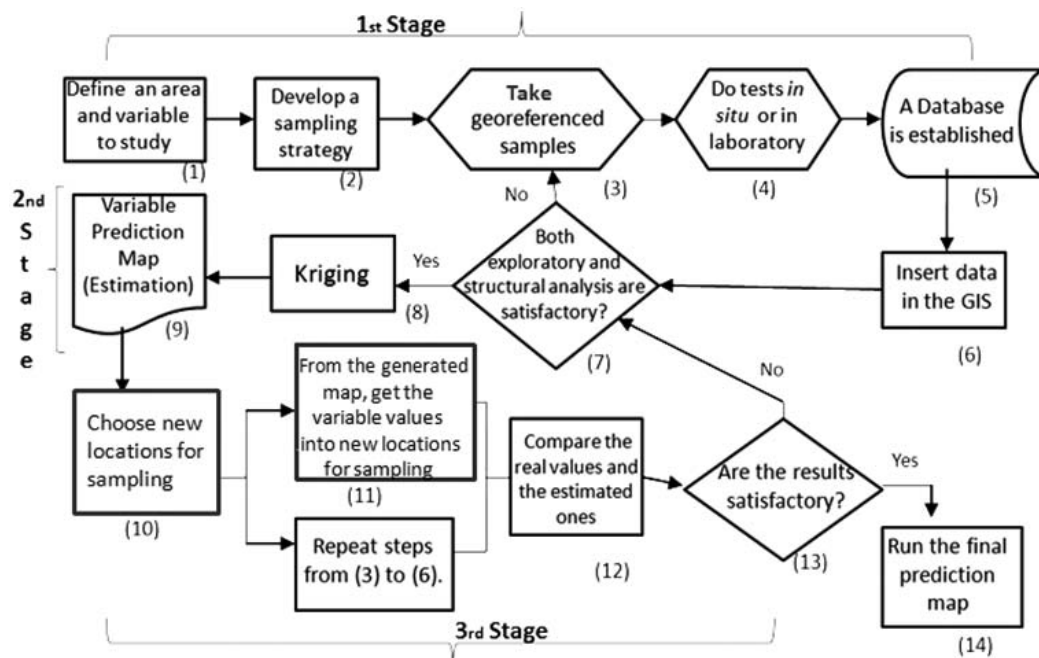
### 2.4. Geostatistics procedures

The procedures adopted for the application of geostatistics approach permit the evaluation, at each stage, the quality of data and the partial results obtained and seeking new solutions to improve the final results. Figure 5 summarizes the step-by-step procedures used to prepare the map for predicting porosity and the different options for certain situations (Camarinha *et al.*, 2008). The steps were organized in three main stages: 1st stage - identifying and preparing data (from step 1 to 5), 2nd stage - analysis of data (step 6 to 9) and 3rd stage - optimization of the process (step 10 to 13).

#### 2.4.1. Definition and preparation of data

After the definition of the study area, *Santa Edwiges* farm, the total porosity was chosen as the variable of soil being analyzed by the present proposals due to two factors: a) easy in the sampling process and laboratory testing and b) association with the infiltration process and water movement in soil.

The next step was the establishment of the database, which originally were constituted of 30 georeferenced samples, in continuation of the field works of Domingos (2004). Each sample is constituted of the following parameters: total porosity, natural and dry specific gravity, moisture content, void ratio and specific gravity of the grains evaluated through the classical soil mechanics approach at



**Figure 5** - Schematic chart proposed for the procedures adopted in the preparation of a map to predict desirable quality (Camarinha *et al.*, 2008).

the Laboratory of Soil Mechanics, UNESP/FEG. All these parameters were obtained at 3 different depths: surface (samples taken between 0 and 20 cm), 50 and 100 cm.

#### 2.4.2. Data analysis

In this study, the establishment of a georeferenced database was made by inserting new points, one by one, using a Geographic Information System (ArcGIS® 9.2 and 9.3 versions) available at the Laboratory of GeoSpatial Analysis (LAGE). Then, the values of various physical indexes known were assigned, among them the porosity, the parameter chosen for this study.

From these data, it was aimed to elaborate a partial map to predict the special distribution of soils porosity and also the error associated with the estimates. This error map is prepared analogously to the prediction of any variable. However, instead of using for each point the known value of the variable, it is used all design points on prediction in order to estimate the values. This error exists due to the fact that kriging considers the influence of neighboring samples to estimate the values in any location; even if there is already a collected sample in that place. Thus, the estimated value is not necessarily equal to the actual value of the field sample. Both maps are made using the extension Geostatistical Analyst, ArcGIS® program. The theoretical framework used in the routine of the program is presented in 0.

It is important to observe that in the steps preceding the drafting of the final prediction map (step 14, Fig. 5), some statistical analysis (step 7) such as verification of the histogram, cross-validation, trend analysis and so on, will not be carried out in detail. This is done because the methodology could become impractical. Then, every time it becomes necessary to draw up a map of preliminary prediction (step 9). Nevertheless, attention will be focused just on the obtaining variograms that are able to verify spatial dependence of the study variable.

#### 2.4.3. Process optimization

The quality of dataset collected is evaluated from the spatial and exploratory analysis (histograms, verification of clusters etc.). Depending on such assessments, one can verify whether the dataset provides sufficient conditions for the generation of reliable maps prediction (Houlding, 2000). If this is not checked, it is possible to use dataset to get information that could guide the development of the next steps of the research. Then, it is possible to devise better strategies from sampling and then avoid the difficulties encountered previously.

Before heading to the final steps which involve the geostatistics, it is necessary that the dataset show a desirable quality in accordance with the reality observed in the field. This assessment is made by comparing the real values of the variable, taken in the field, and the preliminaries values estimated by kriging. Only after achieving the desired

quality, further studies will be directed to deep analysis in order to provide the final maps.

The analyses of this step make possible to verify the prediction error distribution, the existence and representativeness of the variogram and, besides, whether the actual result is consistent with that expected for the variable studied (Camarinha et.al, 2008). For all the preliminary steps were generated experimental semivariograms. However, in this paper, only the semivariograms for the final stage were presented.

### 3. Results and Discussions

#### 3.1. Procedure for the prediction of the porosity maps and the associated error

The analysis started with 30 existed samples collected and tested at the Laboratory of Soil Mechanics, UNESP/FEG. Figure 6 shows the location of each sample within the Santa Edwiges Farm. Its spatial position and the porosity values for each depth (samples taken between 0 and 20 cm, 50 cm and 100 cm) are presented in the table.

Based on this initial database, the first analysis was carried out to produce a map with the prediction of the surface porosity and the associated error of the estimate (Fig. 7). The figure presents the first map generated, in which the darker lines and surfaces represent where there is higher prediction error and higher porosity, respectively.

In this first preliminary map, shown in the Fig. 7(A), the regions near the physical boundary of the study area, especially in the north portion, identify areas with greater uncertainty, for this reason, a new set of soil sampling were carried out in this area as illustrated in Fig. 7(B). These samples also allowed the verification whether the number of samples is representative regarding the different geologic units within the study area.

From the results obtained with these new samples, a comparison was made between the values of porosity estimated by kriging (Fig. 7A) with the actual values determined from laboratory tests (Table 1).

The results shows that only two points (34 and 35), show the real values quite discrepant from those estimated by the kriging method. Considering the size of the study area (12 km<sup>2</sup>) and the low number of samples at this stage, this initial comparison of the data reflects a substantial quality of the interpolation model used.

Although the quality of these preliminary results were acceptable, it was produced another map with the prediction error using the new dataset with 38 sampled points. Maps 1 and 2 in Fig. 8 represent the distribution of the error for both cases analyzed and indicate the region of new soil investigation.

From map 2 in Fig. 8 it is still possible to observe the existence of a region with high error at the northwest portion of the area, which means that the predicted porosity has low accuracy. Based on this observation, it was sought to

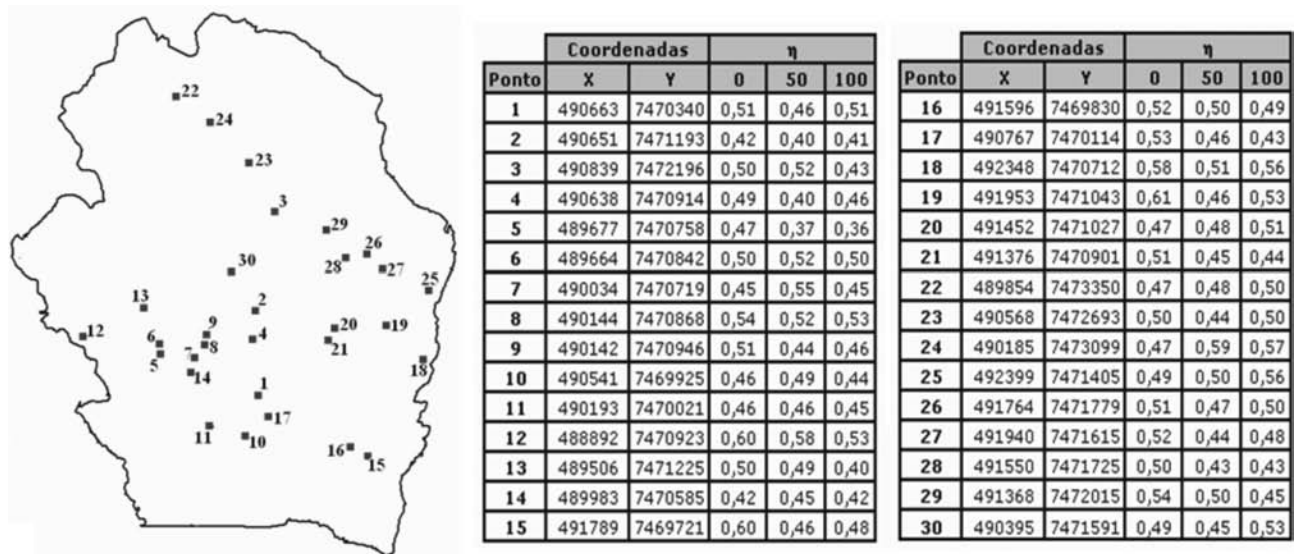


Figure 6 - Location and attributes of the first 30 samples.

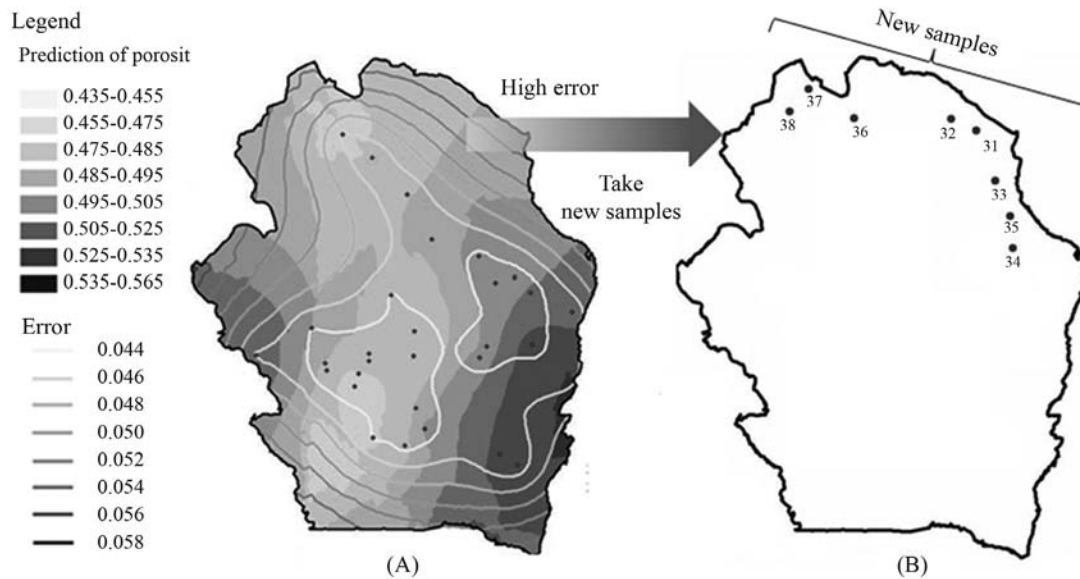


Figure 7 - (A) Prediction of surface porosity and the distribution of error associated; (B) the new locations of soil sampling.

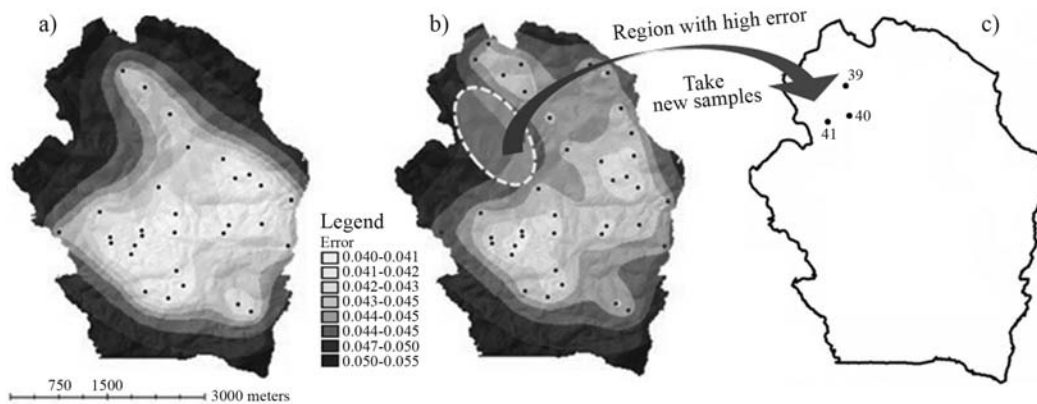


Figure 8 - Maps of error associated with the prediction of surface porosity: (A) database with 30 samples; (B) database with 38 samples. Map (C), location of the 3 new soil samples.

**Table 1** - Comparison between the values of soil porosity obtained by laboratory analysis (Real value) and estimated by kriging (Estimated value).

| Sample | Estimated value | Real value | Real error | Estimated error |
|--------|-----------------|------------|------------|-----------------|
| 31     | 0.490           | 0.474      | 0.016      | 0.033           |
| 32     | 0.495           | 0.520      | 0.025      | 0.048           |
| 33     | 0.495           | 0.465      | 0.030      | 0.061           |
| 34     | 0.510           | 0.414      | 0.096      | 0.188           |
| 35     | 0.505           | 0.420      | 0.085      | 0.169           |
| 36     | 0.482           | 0.489      | 0.007      | 0.015           |
| 37     | 0.475           | 0.459      | 0.016      | 0.034           |
| 38     | 0.492           | 0.477      | 0.015      | 0.031           |

collect three new samples in this region, indicated by the samples 39, 40 and 41 located at the right on the map in Fig. 8.

At this stage of the analysis, the sampling procedure was completed with 41 samples to create the final map. Their spatial coordinates and values of porosity for the three depths analyzed are shown in Table 2. No sampling was made at the extreme southern and western parts of the study area because access difficulties (steep slopes and dense forest).

Before carrying out the final kriging process the quality of data was evaluated in order to determine whether they present a good spatial dependence and characteristics that are consistent with the basic principles of geostatistics.

### 3.2. Final maps of porosity

To create the final maps with the predictions of the porosity for the three different depths, it is necessary to investigate whether the distribution of the porosity variable allows the application of geostatistic methods (structural and exploratory analysis). After this step, it was analyzed if the results of the predicted porosity were consistent with the values from the literature that's correlate porosity regarding geological units.

#### 3.2.1. Histograms

To examine the quality of database, some authors proposed the use of histograms to verify the kind of data distribution (Folegatti, 1996; Houlding, 2000). Folegatti (1996) states that for variogram adjustments, the normal distribution of data is just desirable but not a necessary prerequisite. If the distribution found of data is not normal, but is reasonably symmetrical, it is possible to accept the assumptions necessary for the construction of the semivariogram.

The traditional histogram only shows the frequency of some bands of variable values, for example points having porosity between 0.41 and 0.43, but the distance between samples is ignored. Therefore, although this is not a

**Table 2** - Final dataset with 41 sampling points and their attributes.

| Sample | Coordinates in UTM |         | Porosity ( $\eta$ ) |       |        |
|--------|--------------------|---------|---------------------|-------|--------|
|        | X                  | Y       | Surface             | 50 cm | 100 cm |
| 1      | 490663             | 7470340 | 0.51                | 0.46  | 0.51   |
| 2      | 490651             | 7471193 | 0.42                | 0.4   | 0.41   |
| 3      | 490839             | 7472196 | 0.5                 | 0.52  | 0.43   |
| 4      | 490638             | 7470914 | 0.49                | 0.4   | 0.46   |
| 5      | 489677             | 7470758 | 0.47                | 0.37  | 0.36   |
| 6      | 489664             | 7470842 | 0.5                 | 0.52  | 0.5    |
| 7      | 490034             | 7470719 | 0.45                | 0.55  | 0.45   |
| 8      | 490144             | 7470868 | 0.54                | 0.52  | 0.53   |
| 9      | 490142             | 7470946 | 0.51                | 0.44  | 0.46   |
| 10     | 490541             | 7469925 | 0.46                | 0.49  | 0.44   |
| 11     | 490193             | 7470021 | 0.46                | 0.46  | 0.45   |
| 12     | 488892             | 7470923 | 0.6                 | 0.58  | 0.53   |
| 13     | 489506             | 7471225 | 0.5                 | 0.49  | 0.4    |
| 14     | 489983             | 7470585 | 0.42                | 0.45  | 0.42   |
| 15     | 491789             | 7469721 | 0.6                 | 0.46  | 0.48   |
| 16     | 491596             | 7469830 | 0.52                | 0.5   | 0.49   |
| 17     | 490767             | 7470114 | 0.53                | 0.46  | 0.43   |
| 18     | 492348             | 7470712 | 0.58                | 0.51  | 0.56   |
| 19     | 491953             | 7471043 | 0.61                | 0.46  | 0.53   |
| 20     | 491452             | 7471027 | 0.47                | 0.48  | 0.51   |
| 21     | 491376             | 7470901 | 0.51                | 0.45  | 0.44   |
| 22     | 489854             | 7473350 | 0.47                | 0.48  | 0.5    |
| 23     | 490568             | 7472693 | 0.5                 | 0.44  | 0.5    |
| 24     | 490185             | 7473099 | 0.47                | 0.59  | 0.57   |
| 25     | 492399             | 7471405 | 0.49                | 0.5   | 0.56   |
| 26     | 491764             | 7471779 | 0.51                | 0.47  | 0.5    |
| 27     | 491940             | 7471615 | 0.52                | 0.44  | 0.48   |
| 28     | 491550             | 7471725 | 0.5                 | 0.43  | 0.43   |
| 29     | 491368             | 7472015 | 0.54                | 0.5   | 0.45   |
| 30     | 490395             | 7471591 | 0.49                | 0.45  | 0.53   |
| 31     | 491471             | 7473389 | 0.47                | 0.48  | 0.48   |
| 32     | 491194             | 7473517 | 0.52                | 0.49  | 0.39   |
| 33     | 491673             | 7472839 | 0.47                | 0.56  | 0.51   |
| 34     | 491873             | 7472103 | 0.41                | 0.44  | 0.43   |
| 35     | 491839             | 7472453 | 0.42                | 0.38  | 0.42   |
| 36     | 490129             | 7473522 | 0.49                | 0.49  | 0.57   |
| 37     | 489625             | 7473842 | 0.46                | 0.42  | 0.53   |
| 38     | 489422             | 7473598 | 0.48                | 0.51  | 0.46   |
| 40     | 489707             | 7472633 | 0.45                | 0.51  | 0.55   |
| 39     | 489670             | 7473159 | 0.48                | 0.54  | 0.51   |
| 41     | 489361             | 7472539 | 0.51                | 0.52  | 0.5    |

common practice, it was established relations between the intervals of higher frequency of the histogram and the location of samples which is composed (Fig. 9). This analysis can help identify whether the most similar samples are located close or far apart.

From Fig. 9, it is observed that both results, on the surface as well as at 100 cm depth are consistent with one of the main principles of the geostatistical approach that state that samples taken closer together are more similar on average (ESRI, 2001; Nicol *et al.*, 2003). For the depth of 50 cm, similar samples are scattered throughout the region and are far from each other.

### 3.2.2. Variograms

The analysis was performed aiming the identification of the semivariogram model that best fits the distribution of points. This step is undertaken by using the ArcGIS® software which seeks to establish an approximate semivariogram via the observed distribution. Figure 10 represents this check and, when the parameters that define them are identified, they are highlighted: (a) range, (C) sill and (Co) nugget effects. Therefore, the variogram was fitted by a spherical theoretical curve with a nugget and used for subsequent analysis.

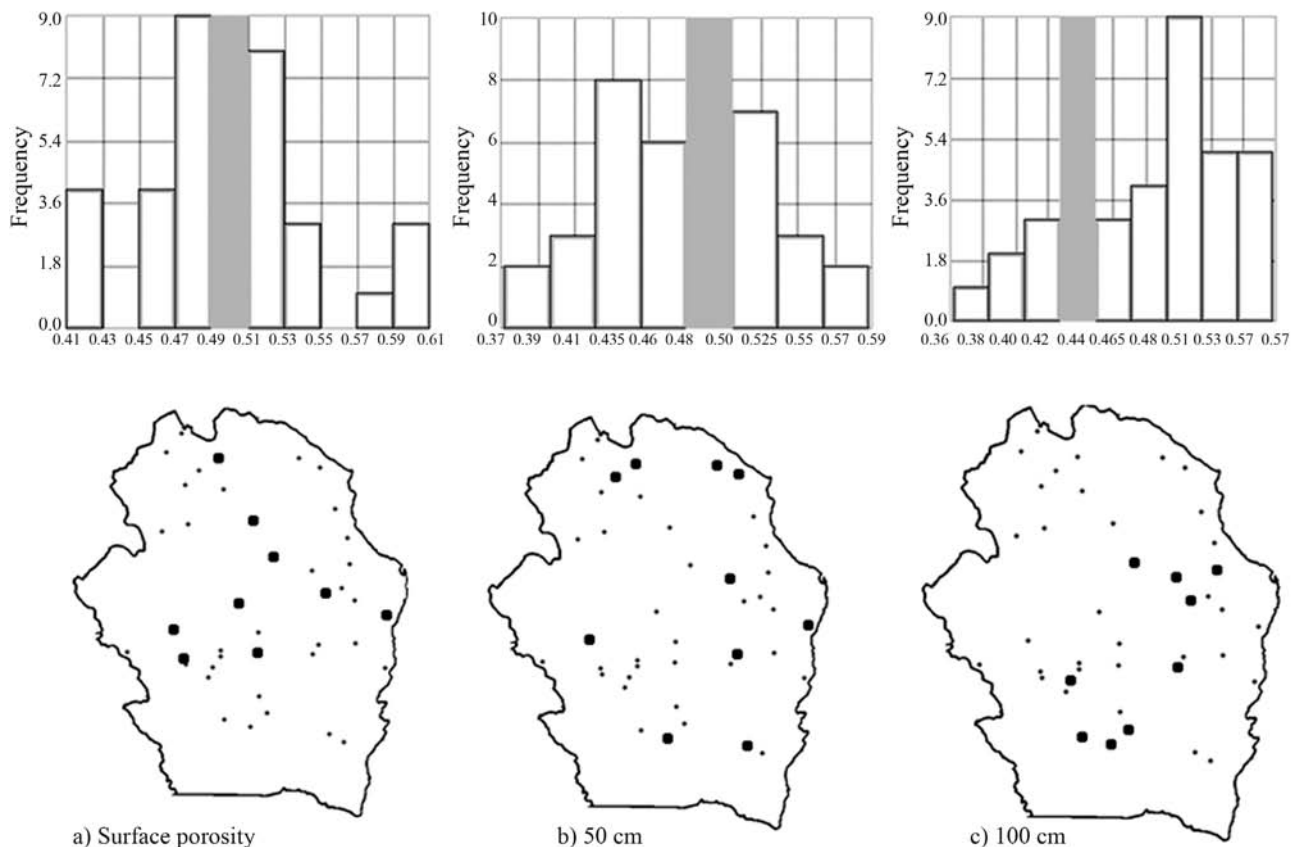
It is possible to observe at Fig. 10 that the data collected on the surface (Fig. 10-a) and at 100 cm depth

(Fig. 10-c) showed a spatial distribution which allowed the adjustment of the semivariogram model. On other hand, the dispersion of the data for the depth of 50 cm (Fig. 10-b) made impossible to adjust any theoretical variogram. For this reason, it was impossible to create prediction maps from kriging method for 50 cm depth with the risk of generating inconsistent results. Therefore, the further investigation was carried out for these two depth, in other words, the kriging analyses were applied to predict surface and at 100 cm depth porosities.

One of the most important steps is to find an theoretical variogram bases on experimental variogram (circular, spherical, gaussian, among others), which best approximates to the observed distribution. From the theoretical variogram chosen, the interpretation of the spatial correlation structure using krigings inferences is made (Camargo *et al.*, 2004; Silva, 2005). Using the *Geostatistical Wizard* tool - which makes up the *Geostatistical Analyst* extension of ArcGIS, it was analyzed the variability in both depths (superficial and 100 cm depth). Thus, it was obtained the variograms presented in Fig. 11 for both depths using the Spherical model as a model for the theoretical variogram.

### 3.2.3. Search neighborhood settings

It is common in practice to specify *search neighborhood* that limits the number and the configuration of the



**Figure 9** - Histograms of the spatial distribution - a) surface, b) 50 cm, c) 100 cm.

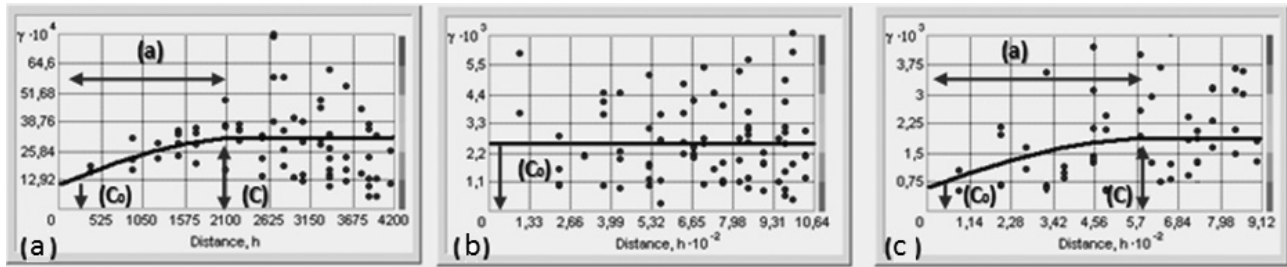


Figure 10 - Variograms for the depths: (a) surface, (b) at 50 cm and (c) at 100 cm.

points that will be used in the predictions. There are two controlling mechanisms to limit the points used: specifying the shape of neighborhood and establishing constraints on the points within and outside the shape (ESRI, 2001).

From the Geostatistical Wizard tool, it is possible to choose the number of surrounding samples that will influence the estimation for a non-sampled location (Neighbors to Include), that is, this number of samples will be included inside the *search neighborhood*. This settings is carried out by the user, generally by trial and error, verifying the number which provides the highest root-mean-square (RMS) and which gives value closer to unity.

For this settings, it is common to divide the *search neighborhood* in sectors because it contributes to a more homogeneous inclusion of the neighbors samples. For the present work, the *search neighborhood* was divided in four sectors in order to always use samples from different portions of the area. It was set to included three samples per

sector, always including at least two samples, because in some locations of the *search neighborhood* can have insufficient samples, requiring its extrapolation (see Fig. 12).

The last adjustments made before the generation of the final maps is the definition of the geometric shape of the *search neighborhood*. Generally, the parameter *range* shapes the *search neighborhood*, which acquires the shape of a circle with radius exactly equals to it. However, it is possible to modify these settings if there are explanations for realize such changes.

For this present research, it was not used a circular shape. A factor of anisotropy for providing an ellipse shape was intentionally used and rotated to 40°. This settings facilitates the use of samples with a higher degree of similarity, reducing the probability of samples with different properties influencing the estimates and, thus, improves the quality of result. These settings were done in accordance

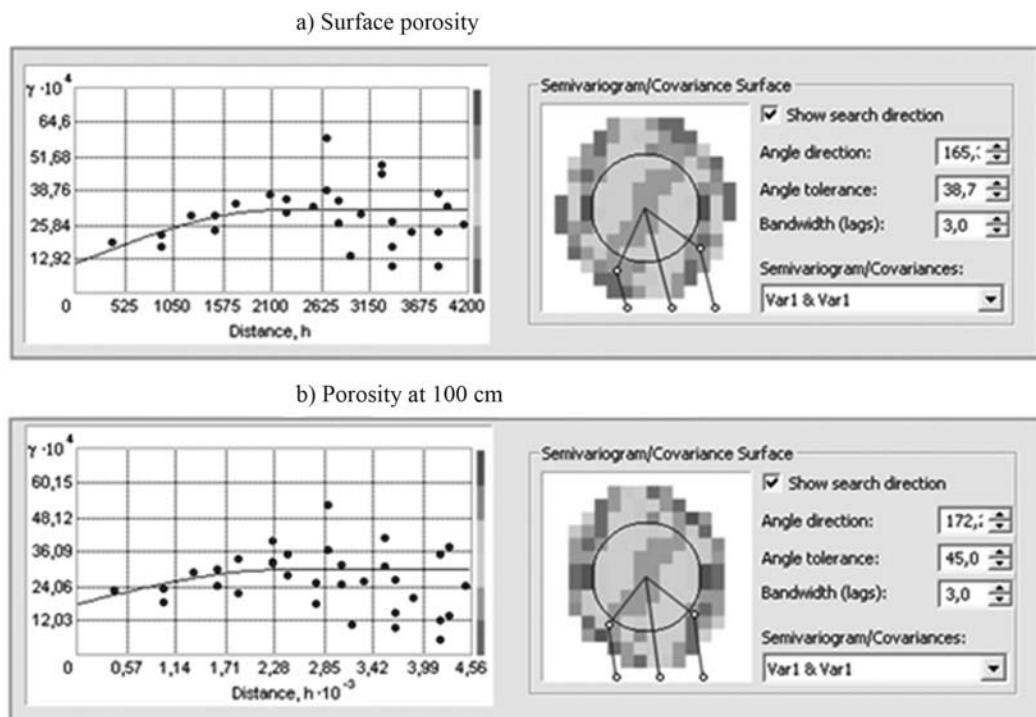
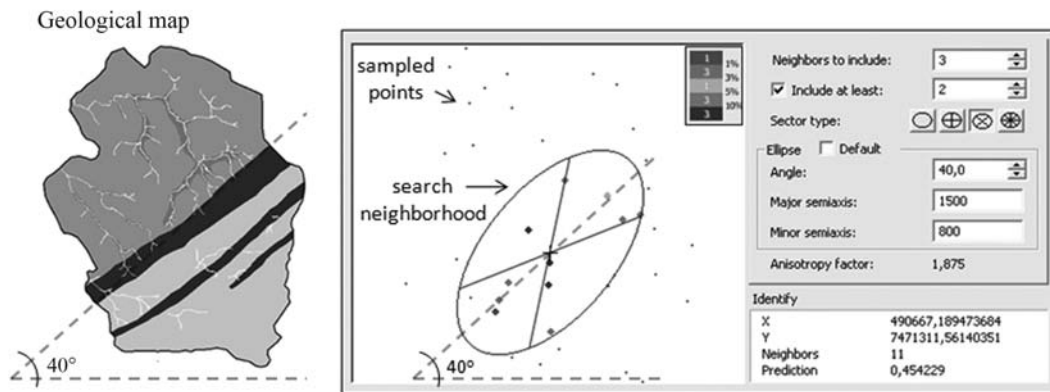


Figure 11 - Directional variograms obtained: a) surface porosity and b) porosity at 100 cm.



**Figure 12** - The Geological Map and the adjustments done to *Search Neighborhood's* shape.

with the Geological Map (Fig. 2), which can be observed a shear zone with same orientation.

Figure 12 shows the ellipse shape of the *search neighborhood* for the surface porosity data, with 1500 m and 800 m for major and minor semi-axis, respectively. The value of 1500 is nearly the same value of the *range* parameter observed in directional variograms (Fig.11).

The same criteria were used for the settings used to porosity data at the 100 cm depth. The only modification was the value adopted by major and minor semi-axis, which was set to 1200 and 600 m, respectively. This reduction was necessary because higher semi-axis values have implied in lower RMS and the values predicted to porosity have presented values nearly equal throughout the area.

### 3.2.4. Porosity prediction by kriging and associated error

The next step of the calculation is the application of kriging with the established parameters such as, the semi-variogram and respective direction, the size of the area search, number of neighbors included. The software ArcGIS® is now able to run the mathematical algorithm to calculate the spatial value of the porosity as presented in Fig. 13 for the two investigated depth.

The kriging method also allows the knowledge of the distribution of the error associated with estimates as explained in section 2.4.2. The quality of the maps for prediction of porosity presented in Fig. 13 depends essentially on the distribution of the error existing in the methodology. Figure 14 shows this distribution error for the two depths investigated. For the two maps, the darker areas indicates higher error which means that the predicted porosity is less reliable at this regions and, on the other hand, lighter areas indicate good quality of the prediction.

### 3.3. Discussion

For geotechnical mapping based on geostatistical analysis, the number of sampling must be sufficient. Increasing the number of samples will result in a better representativeness of the variable on the proposed model, reducing the spread of the error. Unfortunately, there are

some site characteristics, topography, vegetation, which field works become restricted.

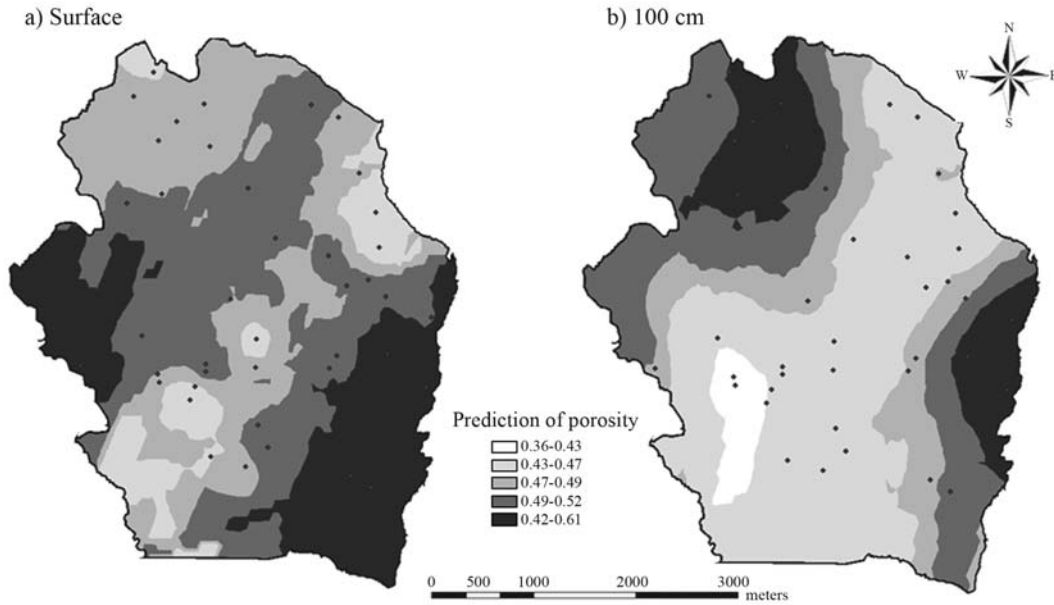
In order to avoid insufficient data or excessive field activities, the proposed methodology permits the identification of areas with some peculiarities which is indicated by the error values. This behavior was observed in points 34 and 35, where the porosities are different from the samples that make up your neighborhood, requiring the increase of sampling process in this region for statistical analysis to become more consistent. This specific area can also characterize a particular geological condition, which must be considered individually.

The geostatistics analyses made from parameters collected at surface and at 100 cm depth gave the spatial dependence which allowed the adjustment of the semivariogram model, allowing the use of kriging of the spatial porosity predictions. This analysis was not possible for the depth of 50 cm due to the scatter of the field data. In this case, a more detail field investigation is required to understand the reason of such variability. Depending on the geomorphology, the variability can be attributed also to the thickness of this intermediate layer and detail investigation must be carried out in small areas.

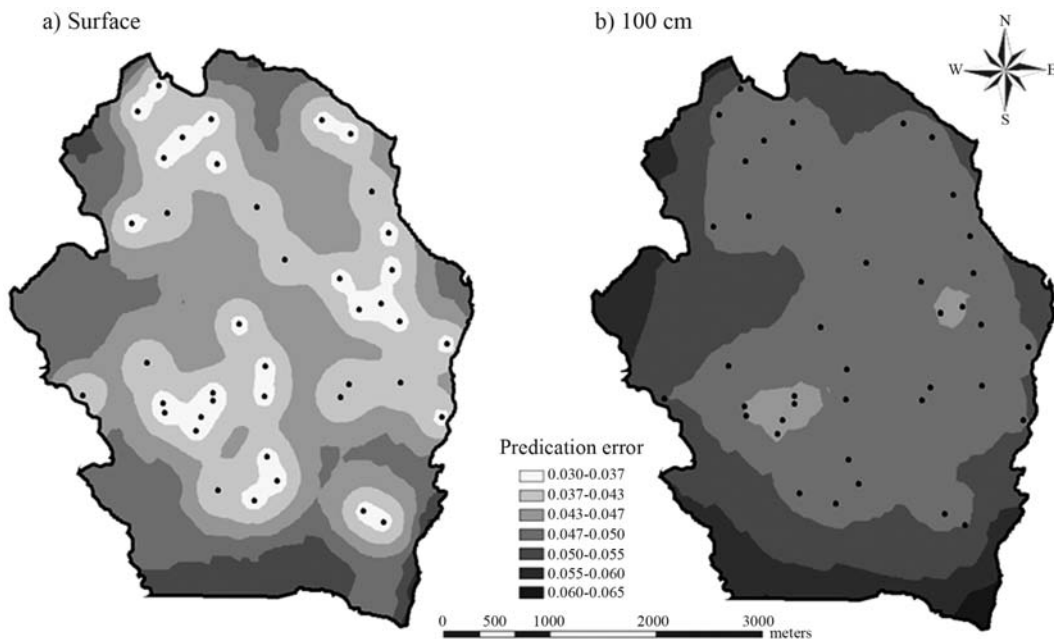
In general, Fig. 14 indicates that the data collected on the surface have better representativeness (lower error) when compared with 100 cm depth. For the surface data, there is a larger amount of samples that have similar values and are spatially close together. This fact represents a greater homogeneity of the surface porosity throughout the study area.

The analysis for 100 cm depth showed lower porosity when compared with the surface porosity. This fact can be explained by the interference of the rooting system of the plants at shallow depth which contributes to more porous soils.

Comparing the predicted maps of porosity with the geological map developed in scale 1:10.000 (Fig. 2), it is possible to make some considerations regarding the results of the geostatistic analyses with the geological units.



**Figure 13** - The final prediction maps generated for the surface porosity (a) and at 100 cm depth (b).



**Figure 14** - Maps of the distribution of error at (a) surface and (b) 100 cm depth.

The geological unit “Granitoids Quebra-Cangalha” is mainly composed of leuco-granite, which occupies the entire southeast study area. The soils from this geological unit have, mostly large amount of coarse material, sand and silt, characteristic of young residual soils, little thick layer, heterogeneous and that would be classified as Cambisols in Pedology. According to the literature, it is expected that these soils have porosity in order of 55% at the surface and reducing with depth. Observing the porosity maps in Fig. 13, it can be observed that the measured and predicted

spatial porosity varies from 53% at the surface to 43% at 100 cm depth. This difference in porosity with increasing depth is one of the factors responsible for the physical conditions that impose restriction to water infiltration process and can induce erosion processes at Santa Edwiges farm (Santos, 2007).

The other geologic unit, is consisted of mylonitic rocks, in which is inserted a shear zone with NE-SW direction, Fig. 2. Due to the rocks formation derived from retro-metamorphism and having many foliation plans, the weath-

ered processes are likely to produce changes in surface and to produce thin soils with a predominant clay fraction. In the other hand, the susceptibility of erosion of these rocks often causes them to be associated with relatively low relief. In fact, this is what is observed in the study area, where the mylonitic zones occupy areas of relatively low height compared to the other geological units such as Granitoids Quebra-Cangalha and the Embu Complex. In the area cropped by the shear zone, the porosity does not show significant variation depending on the depth, varying from 38% to 48% in both cases.

The Embu Complex has a wide variety of rock types, but in the northern portion of the farm there is a predominance of gneissic rocks rich in biotite. The soils of this unit are well developed, mature residual soils with clay texture, thick and homogeneous, which would be classified as Latossolos. The correlation between the sequence of gneissic rocks and predicted maps of porosity indicates an increase of porosity with depth, from 45% to 53%, particularly at the northern end of the study area. This increase might be related to conditions of good drainage and water circulation within these soils, is also an indication that they do not have a high degree of compaction to a meter deep. However, in the eastern portion of the Embu Complex, there was a reverse situation, where the values of porosity were higher at the surface (55%). However, this specific situation should be better assessed by being in a region where the error associated with the estimates was high (Fig. 14), thus requiring a larger number of samples.

These considerations suggest a relationship between the spatial patterns of porosity and units of the geological map presented at scale 1:10.000. With increasing depth, porosity increases in the area of granite rocks, reduces in the area of gneissic rocks rich in biotite and remains relatively unchanged in the fields of rocks strongly deformed (shear zones). In the western portion of the study area, where large errors were found associated with scarcity of samples, the model was not efficient for this analysis, requiring a larger amount of data for future characterization of this area.

#### 4. Final Considerations

This manuscript presents a methodology for correlating soil porosity to the respective geological units using geostatistical analysis techniques, including interpolation data by kriging. Therefore, the comparison between a porosity prediction map - based on geostatistical approach - and a geological map constitutes a different way of comparing these two parameters. Furthermore, the "step by step" method presented in this paper (Fig. 2), in which each sampling procedure was used as a basis for the next step, may be considered a useful method to produce better prediction map for geotechnical parameters.

The proposed methodology permits the identification of areas with some peculiarities, which are indicated by the error values. This analysis helps to avoid insufficient data

or excessive field activities. The behavior observed in points 34 and 35, where the porosities are different from the neighborhood, indicates the necessity of extra soil sampling in order to evaluate if it was a lack of data for geostatistic analysis or a specific area that must be analyzed separately.

For two depths the geostatistics analyses using kriging were possible because the data shows spatial dependence, verified by the semivariogram constructions. This analysis was not possible for the depth of 50 cm due to the dispersion of the field data. Depending on the geology and the geomorphology, the variability can be attributed also to the thickness of this intermediate layer and detail investigation must be carried out in small areas.

It can be noticed that the database used can still be complemented in order to minimize the prediction error and provide a better quality of the results. Even with these matters, the study confirmed the existence of spatial patterns between spatialization of the soil porosity and geology units, which represents a potentiality for correlating with others soil properties, such as hydraulic conductivity, soil water retention curves and erosion potentials.

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# *In Situ* Evaluation of Benzene and Toluene Biodegradation in a Large Block of Gneissic Residual Soil

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**Abstract.** Numerous socio-economic benefits are associated with the oil industry. However, problems cannot be neglected, resulting in many accidents that can occur at any production stage of this industry. A serious problem created by this industry is environmental contamination by hydrocarbons. Gasoline in contact with infiltration water, the aromatic constituents, in particular the BTEX group, partially dissolve being the first contaminants to reach the groundwater. Studies published in the literature indicate that biological degradation is the main mechanism responsible for reducing the concentration of these toxic pollutants. Laboratory studies of this kind have been conducted in small scale columns, where the biological field conditions are not well reproduced. However, studies conducted in a larger scale with structured soil, remaining closer to field conditions, evidenced biodegradation processes of BTEX. This paper presents the *in situ* construction, instrumentation and monitoring of a large rectangular block of undisturbed residual gneissic soil, subjected to a horizontal flow of a contaminant solution composed of water, benzene and toluene to evaluate the biodegradation of these compounds in different points of the block over time. During the period of 85 days, samples of the percolating solution were periodically collected from pre-established monitoring points and the concentrations of benzene and toluene were determined using a coupled technique of gas chromatography and mass spectrometry (GC-MS). Based on the monitoring results, decay in the concentrations of the compounds over time and along the length of the block was observed. These results indicated, consistently with the biological characterization of the soil that showed the existence of microbial degrading activity, the occurrence of biodegradation of benzene and toluene, and, by an extraction process, the retention of these compounds in soil was also verified.

**Keywords:** contamination, soil block, instrumentation, biodegradation, benzene, toluene.

## 1. Introduction

Since the oil crisis in the seventies, Brazil has experienced a strong economic growth that increased oil demand and required large restructuring of the entire chain of oil production, from the discovery of new oil fields and the formation of various petrochemical plants to the increase in distribution networks. As a consequence of this huge and complex structure for production and commercialization, the oil industry deals daily with environmental accidents caused by leaks and spills that occur during the exploration, refining, transportation and storage operations of oil and its derivatives (Corseuil & Marins, 1997; Silva, 2002).

Benzene, toluene, ethylbenzene and xylenes (BTEX), hydrocarbons present in gasoline, show great mobility in water since they are the most water soluble oil compounds,

and they also are the most recalcitrant. Benzene is the most toxic and high concentrations of toluene may be toxic to microorganisms, hampering biodegradation. Thus, in a possible leakage of gasoline, these compounds will be the first to percolate through the soil and reach the water table or aquifer (Alvarez & Illman, 2006).

A variety of techniques (natural attenuation, bioventilation, etc.) has been used to remove dissolved hydrocarbons from soil and groundwater. However, natural attenuation has been noted for its efficiency and low cost. This technique is based on monitoring the environment decontamination by natural environmental factors (diffusion, dispersion, volatilization, sorption, biodegradation and chemical reactions) arising from physical, chemical and biological processes. The biodegradation technique reduces

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Submitted on October 5, 2011; Final Acceptance on November 21, 2012; Discussion open until April 30, 2013.

the mass of contaminants by degradation of organic compounds provided by the action of living microorganisms, which may transform these compounds into harmless ones, limiting the transport of petroleum hydrocarbons to groundwater (Corseuil & Alvarez, 1996; Fetter, 1993; Wiedemeier *et al.*, 1996; Jacques *et al.*, 2007; Corseuil & Marins, 1998; Mazzuco, 2004).

Biological activity is capable of mineralizing or biotransforming organic compounds. Biotransformation consists in the partial degradation of compounds in one or more compounds that may or may not be less toxic than the original substance. Mineralization represents the complete degradation of organic molecules on inorganic substances such as carbon dioxide (CO<sub>2</sub>) and water (Jacques *et al.*, 2007; Corseuil & Marins, 1997).

The degradation of contaminants in soil by microorganisms occurs if: the appropriate degrading microorganisms are naturally present in the soil; the amount of contaminant is not toxic to the microbial population; the environment is adequate and stable so as to allow the survival of the adapted autochthonous microbial population, that is, pH is ideally not excessively acidic or alkaline, or alternately, does not present important fluctuations that could cause impact to the microbiota; moisture is adequate; and there are available nutrients (Mariano, 2006). Thus, depending on the amount of microorganisms in the soil and the type of contaminant, biological activity may be important in reducing the amounts of contaminants and therefore their transport and dispersion. The bioremediation technique has been widely studied and used as a strategic mechanism of transformation of toxic organic compounds (*e.g.* benzene and toluene) into less toxic products (*e.g.* carbon dioxide and water) and the elimination of these compounds from soil and aquifers (Alvarez & Illman, 2006).

A large number of field studies to evaluate soil and groundwater contamination by petroleum products have been developed in the Northern Hemisphere (Borden, Ontario, Canada, Cape Cod, Massachusetts, United States). In Brazil, the field experiments carried out in *Fazenda Ressaçada* (Santa Catarina, Brazil) are noteworthy. However, in all of these cases, the experiments were carried out with sandy soils (Vicente *et al.*, 2009).

Laboratory studies have reported the effects of various environmental factors on the biodegradation of BTEX compounds. Many of these researches have been conducted in micro reactors and soil columns (Mohammed & Allayla, 2000). Studies with bigger microcosms and natural soil give a better idea of real processes as they remain closer to field conditions. Previous research has shown the degradation potential of regular and ethanol-amended gasoline in undisturbed Brazilian soils (Corseuil & Marins, 1998; Österreicher-Cunha *et al.*, 2004, 2007).

As field conditions cannot be fully reproduced in the laboratory, and microbial populations are extremely sensitive and reactive to shifts in their environment, up scaling is

crucial to determine whether microcosms data are representative of actual environmental processes; thus the importance of *in situ* tests.

Since *in situ* studies of biodegradation in tropical soils contaminated by petroleum components are scarce, the primary objective of the research reported herein was to build, instrument and monitor an *in situ* experiment carried out in an undisturbed large block of soil isolated from a residual soil mass of gneissic origin in order to assess biodegradation of benzene and toluene under saturated conditions. The paper describes and discusses the experimental issues associated with the experiments and the results obtained.

## 2. Materials and Methods

The experiment consisted in isolating *in-situ* a large block from a residual soil mass of gneissic origin and subjecting it during a time interval of three months to a permanent flow of water containing dissolved toluene and benzene. During this time, samples of water were collected from the block and concentration of the contaminants measured.

### 2.1. Materials

#### 2.1.1. Soil

##### a) Geotechnical characterization

The experiment was constructed at the Waste Mechanics Laboratory of the Federal University of Viçosa. The block was excavated in the C horizon of a gneissic residual soil profile. The stratigraphy and soil texture were defined based on results of standard penetration tests and visual and tactile analyses performed by Jesus (2009).

The soil was characterized by means of geotechnical tests conducted according to ABNT standards NBR-7181/84 and NRB-6459/84, NRB-7180/84, and NRB-6508/84 - clay fraction mineralogical analysis, chemical and physicochemical analyses and scanning electron microscopy (SEM). According to the Unified Soil Classification System (USCS), the soil was classified as clay of high plasticity (CH). Soil geotechnical properties are listed in Tables 1 and 2.

The saturated soil hydraulic conductivity (ksat) was also determined. For permeability determination, three tests were carried out in the laboratory using a variable head permeameter. The average saturated permeability obtained from these samples is 3.78 E-06 ms<sup>-1</sup>.

##### b) Clay mineralogy analysis

X-ray analysis was conducted with a Rigaku D-Max diffractometer equipped with a cobalt tube (Co-K $\alpha$  radiation) and a graphite curved crystal monochromator operated at 45 kV and 30 mA.

For mineralogical analyses, soil samples were obtained from the C horizon of a gneissic residual soil profile. The X-ray analysis of the soil clay fraction was performed

**Table 1** - Soil grain size distribution and Atterberg limits.

| Grain size distribution (%) |      |      |        | Atterberg limites (%) |                    |                    |
|-----------------------------|------|------|--------|-----------------------|--------------------|--------------------|
| Clay                        | Silt | Sand | Gravel | <sup>1</sup> $w_L$    | <sup>2</sup> $w_p$ | <sup>3</sup> $I_p$ |
| 58                          | 14   | 28   | 0      | 66                    | 32                 | 34                 |

<sup>1</sup>  $w_L$  – liquid limit; <sup>2</sup>  $w_p$  – plastic limit and <sup>3</sup>  $I_p$  – plasticity index.

**Table 2** - Soil physical indexes.

| $G_s$              | $\gamma_n$         | $\gamma_d$         | n    | e    | S     | w     |
|--------------------|--------------------|--------------------|------|------|-------|-------|
| $\text{kN m}^{-3}$ | $\text{kN m}^{-3}$ | $\text{kN m}^{-3}$ |      |      | (%)   | (%)   |
| 27.77              | 15.97              | 12.87              | 0.54 | 1.16 | 58.42 | 24.49 |

$G_s$  - solids unit weight;  $\gamma_n$  - soil natural unit weight;  $\gamma_d$  - soil dry unit weight; n – porosity; w – water content; e – void ratio; S – saturation degree.

in two different types of samples: (i) prepared with natural clay by the paste method for orientation of the clay minerals; (ii) prepared after treating the clay to remove iron oxides, to improve the identification of clay minerals and/or aluminum oxides, possibly present in the soil sample. This analysis was performed on two soil samples.

Iron oxides were removed with sodium bicarbonate citrate dithionite, according to the methodology described by Mehra & Jackson (1960). The evaluation of the diffractograms resulting from X-ray analysis of the clay fraction before and after the removal of iron oxides allowed defining the clay soil as consisting of kaolinite (Ka) and gibbsite (Gb) with traces of goethite (Gt) and hematite (Hm).

#### c) Scanning electron microscopy (SEM)

A microphotograph of a deformed soil sample was produced with a scanning electron microscope (SEM) in order to determine the chemical elements present in soil block. Table 3 presents concentration percentages of the chemical elements found in five points of the analyzed soil sample. The results show higher percentages of oxygen (O), aluminum (Al), iron (Fe) and silica (Si).

#### d) Chemical and physicochemical properties

Chemical and physical-chemical properties of the soil are shown in Table 4.

**Table 3** - Concentration percentage of chemical elements found in soil sample.

| Point | O     | Al    | Si    | K    | Ti   | Fe     |
|-------|-------|-------|-------|------|------|--------|
| 1     | 38.54 | 25.06 | 28.04 | 0.46 | ND   | 7.89   |
| 2     | ND    | ND    | ND    | ND   | ND   | 100.00 |
| 3     | 32.14 | 19.03 | 18.63 | ND   | 1.70 | 28.50  |
| 4     | 55.18 | 20.50 | 22.11 | ND   | ND   | 2.21   |
| 5     | 41.07 | 16.80 | 23.24 | ND   | 1.40 | 17.50  |

ND – Not detected.

**Table 4** - Chemical and physicochemical analyses of the soil.

| P                       | K                       | Zn                      | Fe                      | Mn                      | Na                      | Cu                      | Cr                      | Ni                      | Cd                      | Pb                      | pH   |
|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|------|
| ( $\text{mg dm}^{-3}$ ) | ( $\text{mg dm}^{-3}$ ) | ( $\text{mg dm}^{-3}$ ) | ( $\text{mg dm}^{-3}$ ) | ( $\text{mg dm}^{-3}$ ) | ( $\text{mg dm}^{-3}$ ) | ( $\text{mg dm}^{-3}$ ) | ( $\text{mg dm}^{-3}$ ) | ( $\text{mg dm}^{-3}$ ) | ( $\text{mg dm}^{-3}$ ) | ( $\text{mg dm}^{-3}$ ) |      |
| 0.80                    | 37                      | 1.28                    | 22.50                   | 5.00                    | 10.20                   | 0.58                    | 0.32                    | 0.19                    | 0.0                     | 0.06                    | 5.29 |

$\text{CEC}_{(m)}$  - Cation Exchange Capacity

$\text{CEC}_{(e)}$  - Effective Cation Exchange Capacity

SB - Sum of Exchangeable Bases OM - Organic Matter (OM = OC x 1.724 - Walkley-Black OC - Organic Carbon

Considering the mineralogical composition of the clay particles (in high percentage) and its low organic matter content, the studied soil should have little capacity of retaining benzene and toluene or, at least, of retarding their migration.

According to the values showed in Table 4, the natural soil presents the following characteristics:

- Acidic pH, which is generally associated with the presence of aluminum and manganese in toxic concentrations, and low levels of cations such as  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ . The acidity may decrease the microbial population of organic matter decomposers, depending on soil characteristics and environmental conditions.
- Low percentage of organic carbon. The organic matter content is the soil characteristic that most influences sorption of hydrocarbons (Jacques *et al.*, 2007).
- The amount of inorganic contaminants (zinc, copper, nickel, iron, chromium, cadmium, lead and manganese) is within the thresholds established by the Soil and Groundwater Guiding Values (CETESB, 2009).
- According to calcium and magnesium contents, the soil is classified as a low fertility soil by the Soil Fertility Commission of the State of Minas Gerais - CFSEMG (1999).
- The soil presents low effective cation exchange capacity, according to the chemical classification of the CFSEMG (1999).

#### e) Microbiological analysis

Microbiological activity in soil was determined on five soil samples collected in an representative area of the experiment, according to the methodology for measuring hydrolysis of fluorescein diacetate (FDA), following a protocol adapted from the Adam and Duncan (2001) and Green *et al.* (2006) methodologies (Österreicher-Cunha *et al.*, 2007).

The results summarized in Table 5 show low microbial degrading activity considering that the samples were retrieved from a superficial soil in area covered by vegetation, and that the soil contains more than 50% of clay in its composition. Activity levels detected (mean of  $0.829 \text{ mg fluorescein.min}^{-1}.\text{g}^{-1}$ ) were close to those found in stable undisturbed subsurface Brazilian soils and in stabilized contaminated soil undergoing biodegradation of organic compounds (Österreicher-Cunha *et al.*, 2007, 2012).

**Table 5** - Total degrading activity.

| Sample | $\mu\text{g fluorescein.g}^{-1}$<br>$\text{soil.min}^{-1}$ | Standard<br>deviation | Depth (m) |
|--------|------------------------------------------------------------|-----------------------|-----------|
| A      | 0.831                                                      | 0.162                 | 0.40      |
| B      | 0.836                                                      | 0.515                 | 0.40      |
| C      | 0.928                                                      | 0.299                 | 0.40      |
| D      | 0.607                                                      | 0.000                 | 0.50      |
| E      | 0.943                                                      | 0.093                 | 0.45      |

#### 2.1.2. Contaminant solution

An aqueous solution prepared from the mixture of benzene, toluene and water from an artesian well was used in the experiment. The concentration of each compound in solution was set within the limit of solubility of each (benzene:  $1800 \text{ mg L}^{-1}$  and toluene:  $500 \text{ mg L}^{-1}$ ).

Forty five liters of contaminant solution were required per day to feed the upstream reservoir. This volume was determined based on the water flow in the block for steady state conditions.

The water from the artesian well showed neutral pH (6.94) and a high concentration of dissolved oxygen ( $5 \text{ mg O}_2 \text{ L}^{-1}$ ) both favorable to microbial survival; low concentration of potassium, phosphorus and nitrogen; and low concentrations of chemical substances (*e.g.* calcium, magnesium, aluminum, lead, iron, cadmium, silver and sodium). The water was considered appropriate to perform the experiment.

## 2.2. Methods

### 2.2.1. The block construction

The block subjected to the experiment is prismatic in shape having dimensions 1.5 m long; 0.60 m high and 1.0 m wide. Features of the block geometry, lay-out, location, instrumentation and constructive details are shown in Figs. 1 to 9.

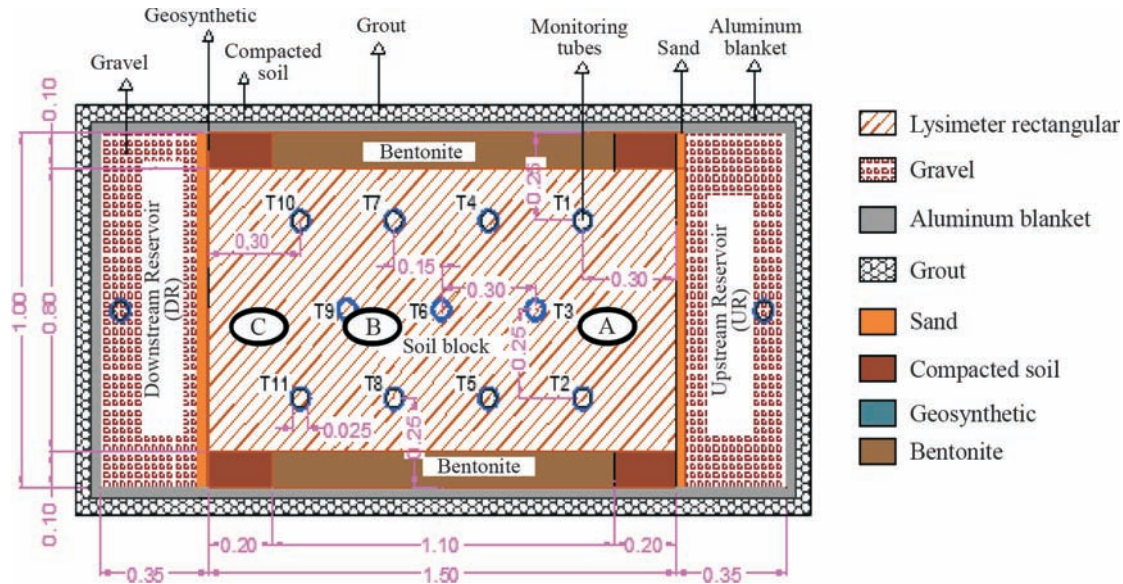
Figure 1 shows the covered area where the experiment was carried out.

The construction of the undisturbed block started with the excavation of its lateral faces using appropriate tools. The lateral faces (length direction) were sculpted with dimensions 1.0 m high and 2.20 m long. In sequence, the block was involved with paraffin to retain its natural water content (Fig. 4).

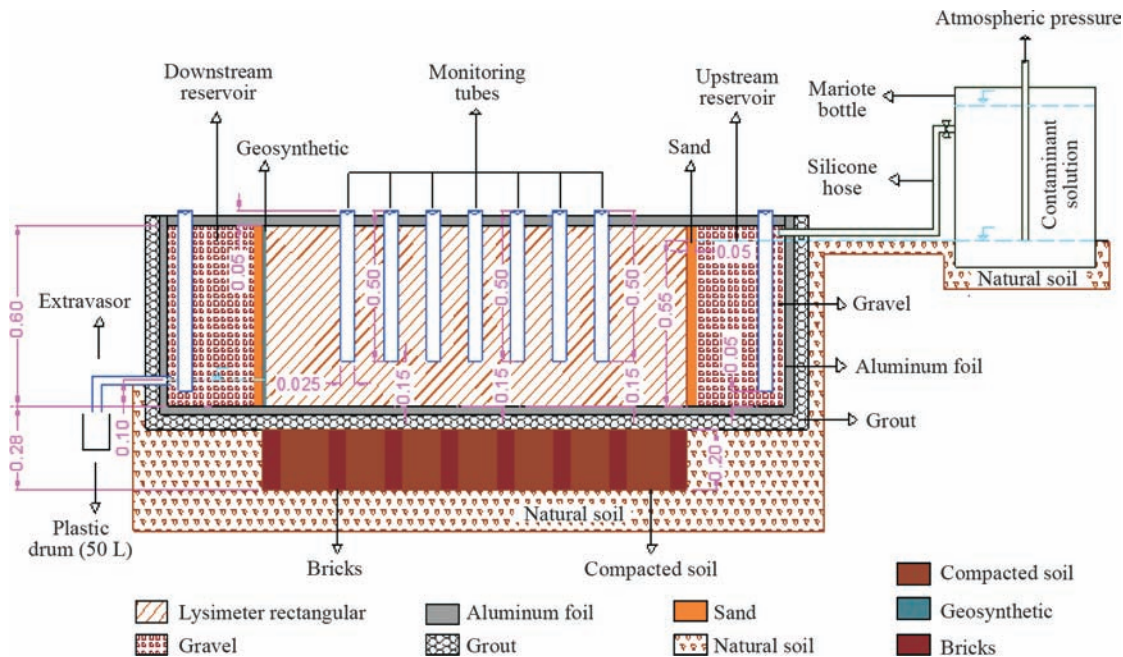
The lateral faces (width direction) remained in contact with the ground until the end of the bottom excavation. Later these faces were excavated for the construction of the reservoirs.



**Figure 1** - Covered area where the soil block was constructed.



**Figure 2** - Plan view of the block detailing the arrangement of monitoring tubes in the block and the upstream and downstream reservoirs.



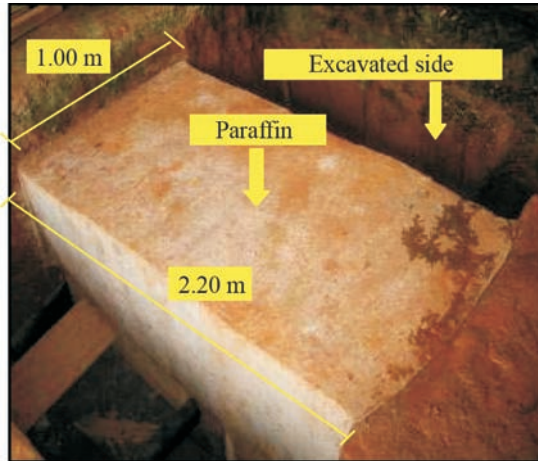
**Figure 3** - Longitudinal section showing the arrangement of tubes, tanks and Mariotte bottle.

The bottom of the block was then progressively excavated in two stages. In the first stage 0.40 m of the bottom part of the height was removed, leaving it with a depth of 0.60 m. Using appropriate tools, the base was trimmed as near as possible of a horizontal plan. At each stage, the soil was removed, an aluminum foil was, step by step, positioned at the bottom face, and a wood panel (0.10 m high, 1.10 m wide and 1.10 m long) supported by bricks, was placed below and the empty space left was filled with a cement mass (grout). Once the grout was dry, the pre-fabri-

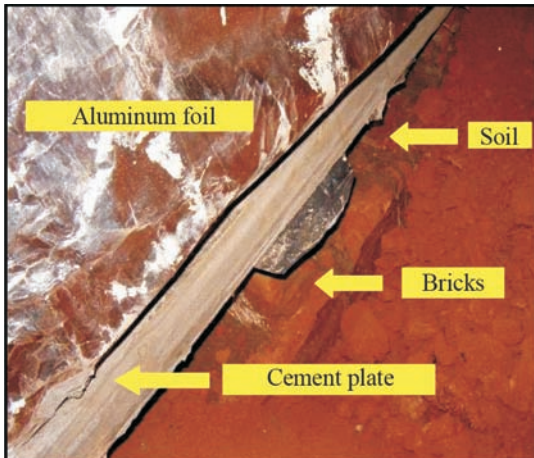
cated wood panel was removed. The cement layer below the bottom of the block was supported by bricks and the void space between the bricks filled with soil as shown in Figs. 3 and 5.

The upstream and downstream reservoirs, 0.35 m long, were dug in the sequence. As a consequence, the block length was reduced to 1.50 m. After the excavation of the upstream and downstream reservoirs the block was partially wrapped with aluminum foil as seen in Fig. 6.

In order to stiffen the block, cement walls, 0.10 m thick, were built around it. These walls were also covered with aluminum foil to avoid sorption of hydrocarbons by the cement, by the time the block was contaminated by the benzene and toluene solution (Fig. 7).



**Figure 4** - Side excavation and block involved with paraffin.



**Figure 5** - Arrangement of materials at the base of the block.

In order to avoid preferential flow between the soil and the aluminum foil on the side walls, a narrow strip of soil, 0.10 m wide, was excavated and filled with soil and bentonite as shown in Fig. 8.

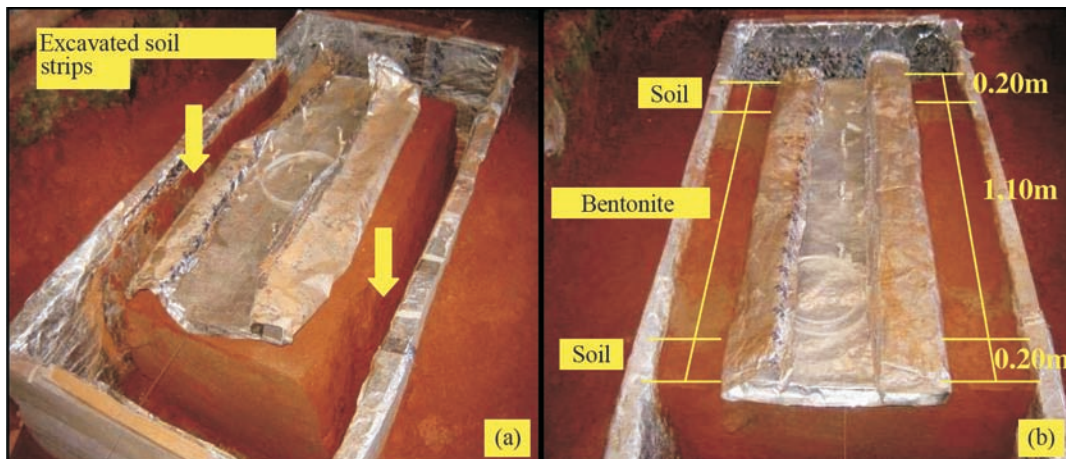
In order to offer extra support to the side walls of the block, the empty space behind them was filled out with compacted soil. In sequence, both reservoirs were filled



**Figure 6** - Excavation of the upstream (UR) and downstream (DR) reservoirs.



**Figure 7** - Side cement walls.

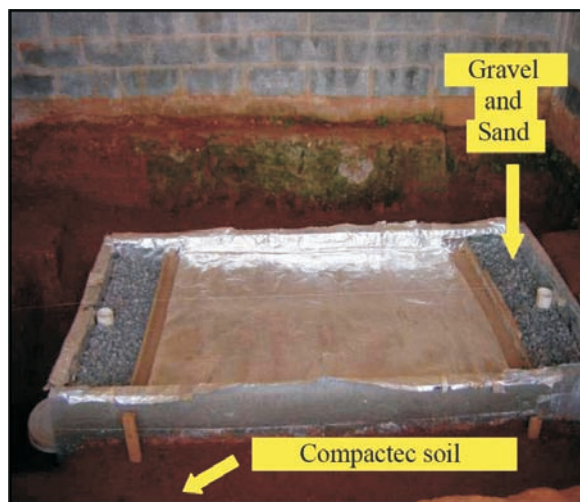


**Figure 8** - (a) Excavated soil strips; (b) Compacted soil and bentonite along the length of the block.

with gravel to avoid disaggregation of soil particles in the block due to moisture. A 0.05 m wide layer of sand was added to avoid soil clogging and damage of the soil surface by the gravel. In the downstream reservoir, between the block face and the sand layer, a geosynthetic was placed to function as a filter, preventing soil particles to enter the reservoir. An overview of the soil block after construction is presented in Fig. 9.

### 2.2.2. Monitoring tubes

Eleven monitoring tubes, one inch in diameter and fifty centimeters in length, made of aluminum were installed along the length of the soil block, 0.15 m from the base (Fig. 10).



**Figure 9** - Overview of soil block showing the final state of both reservoirs.

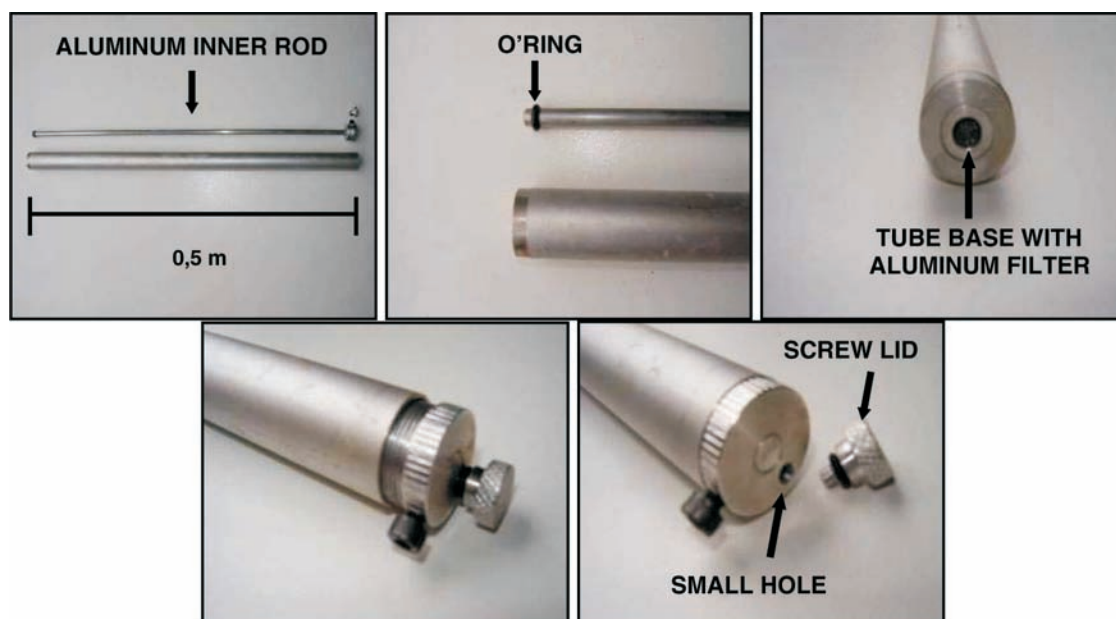
Each monitoring tube had a threaded lid at the top and an aluminum filter at the base to prevent solid particles entering it. Attached to the lid, there was an aluminum inner rod with an o-ring at its tip that controlled the entrance of the contaminant solution through the base. The effluent was removed from the tubes through a hose which was introduced in a small hole (to minimize volatilization of compounds during sampling), with a screw lid.

A metal auger, 0.7 m long and one inch in diameter, was used to drill holes at the points where the monitoring tubes were installed in the soil block, as illustrated in Fig. 11.

In order to maintain a constant level of the contaminant solution in the upstream reservoir, a 50 L stainless steel container was installed to function as a Mariotte bottle. This bottle was connected to the upstream reservoir through a silicone hose, as shown in Fig. 12. The tests were carried out maintaining constant heads both in the downstream and upstream reservoirs.

Figure 12 shows the tube feeding the upstream reservoir and the tube exiting the downstream reservoir. The positions of the extravasation and feed tubes are shown in Fig. 3. The hydraulic head in the upstream reservoir was maintained at 0.55 m above the bottom surface of the block while in the downstream reservoir it was kept at 0.10 m above the bottom surface of the block, resulting in a hydraulic gradient of  $0.30 \text{ m m}^{-1}$ .

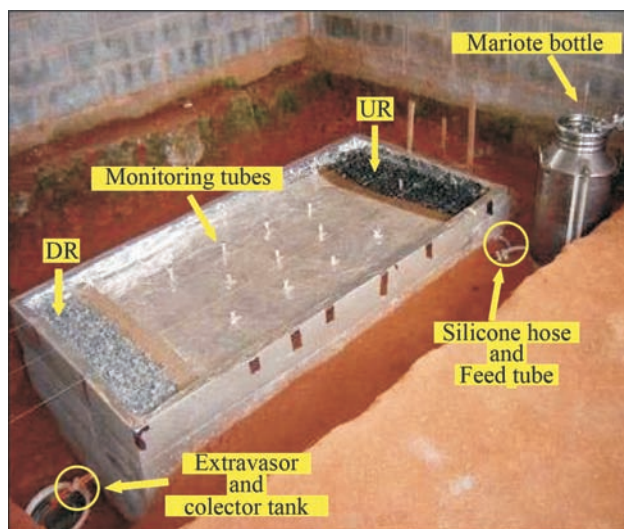
Amongst the difficulties encountered during the construction of the soil block, those considered most relevant were the construction of a cement “box” around it and also, the interruption of preferential flow along the length of the block, only achieved after the use of a compacted layer of bentonite in a strip of 0.10 m.



**Figure 10** - Details of aluminum tubes.



**Figure 11** - Installation of monitoring tubes.



**Figure 12** - Overall view of the experiment.

### 2.2.3. Testing procedure

After finishing the construction and instrumentation of the experiment, the upstream reservoir was filled with water, starting the saturation process of the block with pure water (no contaminant at this stage). During a period of three months, the level of water in the monitoring tubes was measured and the water levels in the block were verified.

The measured average flow rate in the block was of 45 L a day. After the saturation period with water, the up-

stream reservoir was filled with the contaminant solution and the Mariotte bottle was fed daily with the contaminant solution. It was guaranteed that the continuous flow of the solution along the block was not interrupted. The experiment was monitored during three months.

The collection of effluent samples from monitoring tubes was performed by applying suction with the aid of a syringe coupled with a hose that was inserted in the tubes. A volume of 15 mL of the contaminant solution was collected and equally distributed in three vials. The samples were then sent to the laboratory to be analyzed by gas chromatography (Fig. 13).

### 2.2.4. Chemical analysis

The methodology used to extract and quantify the monoaromatic hydrocarbons samples of contaminated soil was developed by Fernando (2010), adapted from Melquiades *et al.* (2006).

The concentrations of benzene and toluene in the contaminated soil were quantified after solvent extraction and static headspace injection into a gas chromatographer equipped with a flame ionization detector (SHIMADZU GC-MS QP 5000), as detailed in Fernando (2010).

Soil samples were collected 83 days after contamination, 0.50 m below the soil block surface, to undergo chemical analyses.

The concentrations of toluene and benzene in the contaminated water were obtained with a SHIMADZU gas chromatographer coupled to a mass spectrometer, using the headspace technique. These analyses were performed at the



**Figure 13** - (a) Introduction of the hose in the monitoring tube; (b) Injection of the contaminated solution in the vial; (c) Sealed vials.

Laboratory of the Chemistry Department at of the Viçosa Federal University according to the methodology proposed by method EPA 8260B.

In the field, 5 mL of contaminated water samples were collected in a silicone lid vial. After arriving in the laboratory, the samples were incubated at 70 °C for 30 min to release volatile compounds. Once removed from the incubator and allowed to cool for 1 min, the manual injection of the volatiles present in the headspace was conducted. For the chromatographic separation, 2 mL of sample was injected with a syringe (HAMILTON). The identification of the pick of each compound was accomplished by the comparison with the time of retention of the peaks in the pattern of benzene and toluene. For the integration of the area of the peaks, each chromatogram was integrated individually, through the Software Labquest Chromatography it Dates System (Wang *et al.*, 2002).

#### 2.2.5. Statistical analysis

Relations between variables were analyzed at 5% level of significance by testing of variance (multi-factor ANOVA). Tests were performed with OpenStat®.

### 3. Results and Discussion

#### 3.1. Effluents analysis

Concentrations of benzene (B) and toluene (T) measured in the contaminant solution of the upstream reservoir remained around 300 mg L<sup>-1</sup> throughout the experiment. Concentrations in the effluent collected from the tubes in the monitored points are presented in Figs. 14b to 14m. Results show, at first, stable concentrations then a trend of decreasing concentrations of benzene and toluene in time in all wells, which may be attributed to retention in soil, volatilization and biodegradation, as discussed further on. The time values in the horizontal axis correspond to the number of days after contamination started (on September 1<sup>st</sup>, 2009).

Graphs in Fig. 14 show stable B and T amounts in upstream and downstream reservoirs; as they were filled with sand and gravel, it was not expected that the granular material would retain significant amounts of these compounds, besides the microbial activity being probably low (not measured). In monitoring wells, B and T amounts decreased significantly only after 45 days, which may correspond to the stabilization of biodegrading activities after the lag time required for the microbiota to adapt. Indeed, besides the natural microbial adaptation, microbial cells may be carried with water flow from the upstream half of the block, increasing biomass and thus rates of biodegradation in its downstream half. Also, lower concentrations found in downstream wells may be due to cumulative retention of contaminants in soil with time as well as to higher biodegradation. The lowest amounts are found in wells 10 and 11, corroborating this hypothesis. The greater the dis-

tance between the monitoring tubes and the upstream reservoir, the lower the concentration of each compound, which can be attributed to the processes of biodegradation and soil sorption, and/or volatilization.

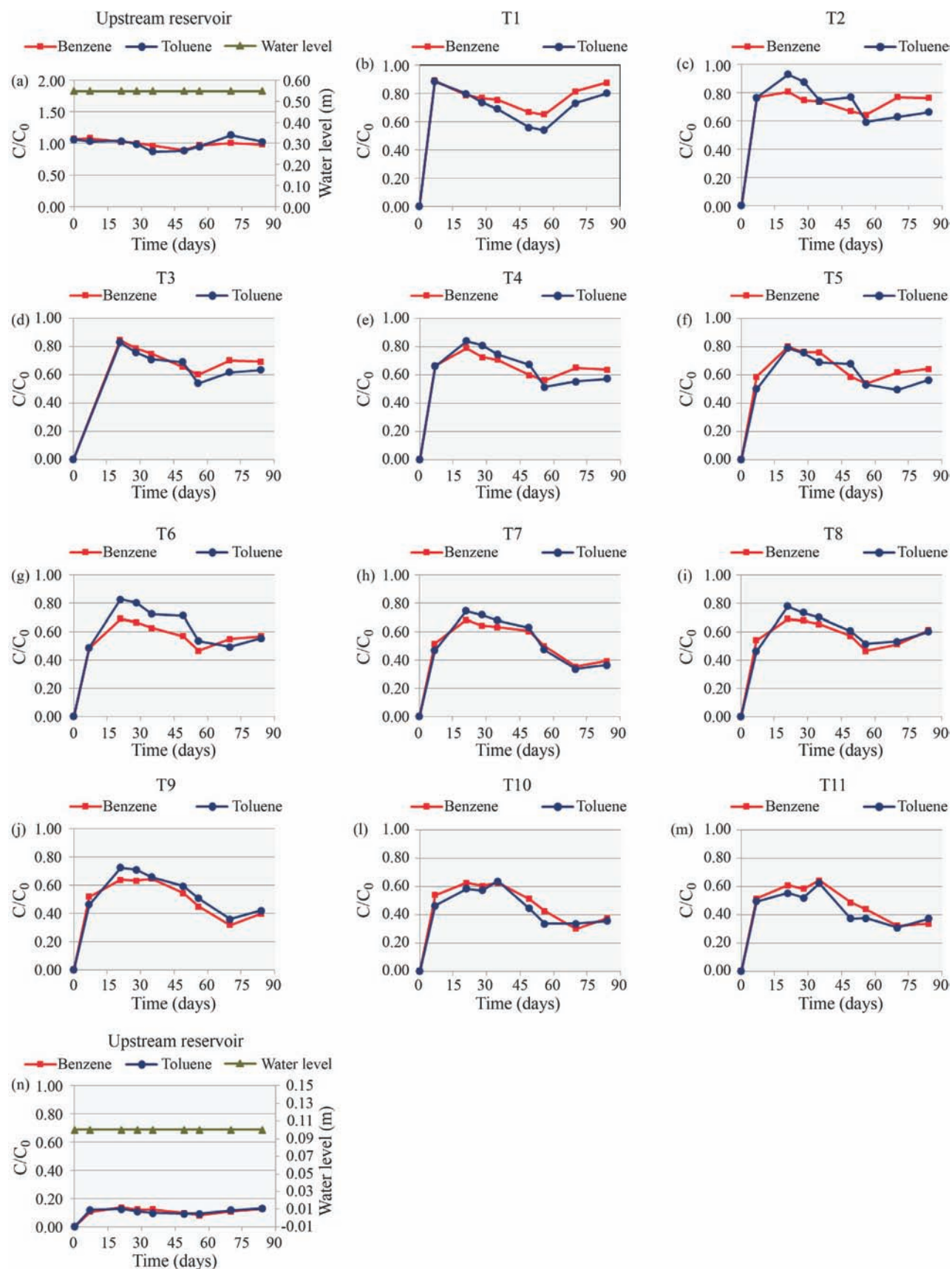
Statistical analysis shows that contaminants concentrations vary significantly in time in both reservoirs and in each monitoring well ( $p < 0.05$ ).

The trend of constant concentration of benzene and toluene in the effluent from the 75<sup>th</sup> day on was expected, considering that the flow regime became permanent. It is possible that the soil reached its maximum capacity of retention of the compounds. As for biodegradation, according to the kinetic model of Monod (1949), a maximum rate followed by a decrease of substrate utilization by microorganisms is expected. However, biological systems present a more complex behavior. The onset of new environmental conditions, as in this case, saturation then contamination, requires an adaptation of the microbial population in order to first survive the impacts and then resume growth and development, which can be assessed by measuring microbial total activity. In the present experiment, the soil block was first saturated and contamination happened three weeks later, when the microbial population would be expected to have already adapted to saturated conditions. An initial impact on microbial activity is generally observed with BTEX contamination (Fernando, 2010; Österreicher *et al.*, 2007). A lag time before activity levels start to increase again corresponds to the adaptation of the microbiota: new strains are selected which are able to survive and use the contaminant as carbon source for survival. With the establishment and the stabilization of biodegradation, lower amounts of contaminant are expected in downstream monitoring wells.

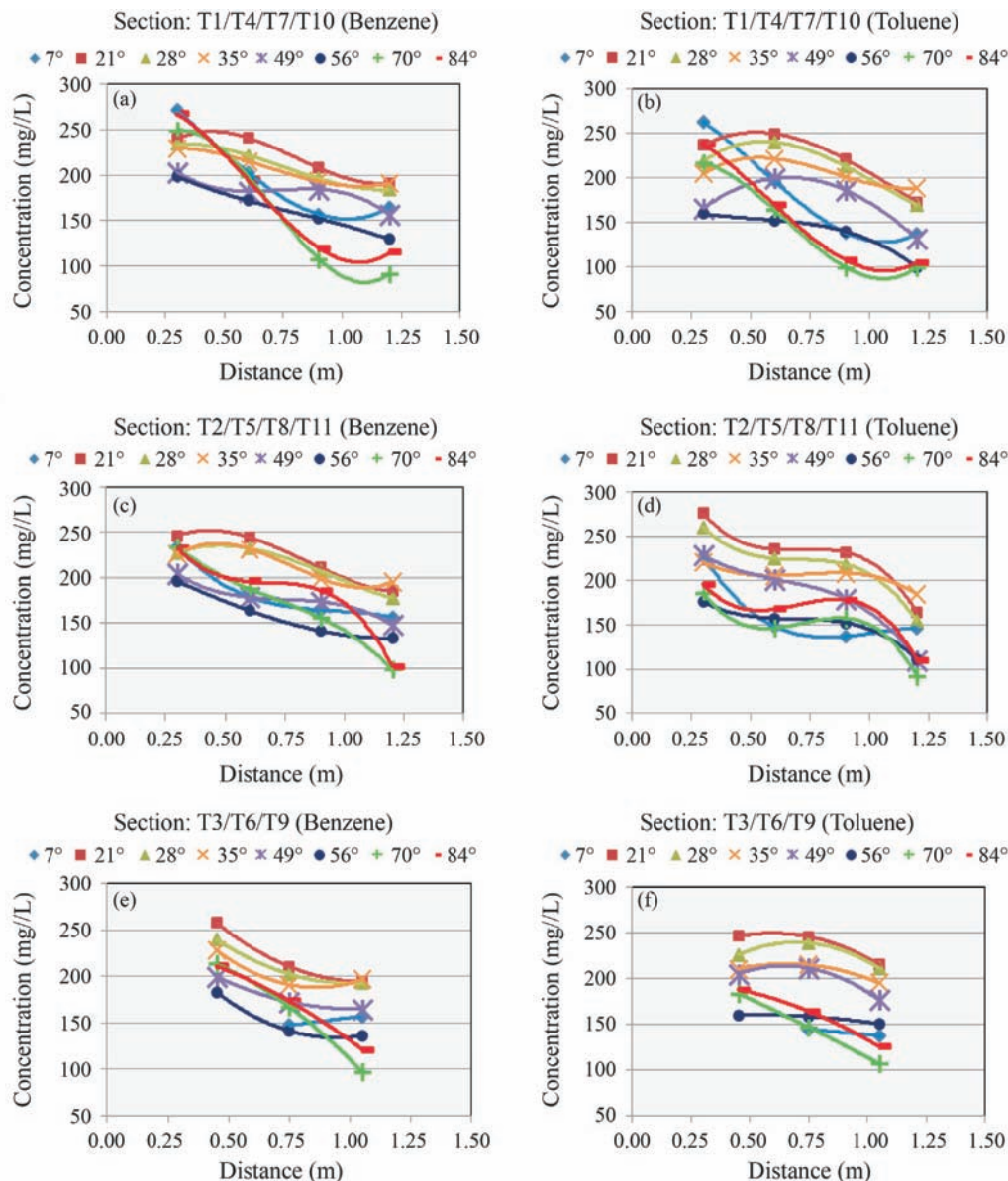
Also, according to the graphs of Fig. 14, concentrations of benzene and toluene showed a similar behavior over time. In most cases, benzene concentration was slightly lower than that of toluene, which could be related to its greater solubility and, therefore, higher bioavailability to microbial attack. However, statistical analysis of results indicates that B and T concentrations are not significantly different in water ( $p < 0.05$ ). From the 49<sup>th</sup> day on, though, a more accentuated fall in the amounts of toluene is observed.

Volatilization was greatly reduced as the block was covered with aluminum foil. Besides, volatilization from saturated soil is also reduced by contaminants dissolution in soil water (Delle Site, 2001).

Figure 15 shows B and T concentrations throughout the experiment along the well lines. At the end of the first week, contaminants are found mostly at the entrance of water flow, in high amounts upstream. From the 21<sup>st</sup> till the 35<sup>th</sup> day, contaminants appear more evenly spread in the whole block, already showing some decrease in concentrations. Samplings on the 49<sup>th</sup> and 56<sup>th</sup> days present a significant decrease in contaminants amounts, more so downstream where probably aerobic biodegradation is still taking place. Lower concentrations next to the source can



**Figure 14** - Benzene and toluene relative concentrations in each monitoring point over time. (a) Upstream reservoir; (b) T1; (c) T2; (d) T3; (e) T4; (f) T5; (g) T6; (h) T7; (i) T8; (j) T9; (l) T10; (m) T11; (n) Downstream reservoir.



**Figure 15** - Variation of Concentration of the compositions benzene and toluene in the monitoring tubes vs. Distance of the tubes from upstream reservoir. (a) Section: T1/T4/T7/T10 (Benzene); (b) Section: T1/T4/T7/T10 (Toluene); (c) Section: T2/T5/T8/T11 (Benzene); (d) Section: T2/T5/T8/T11 (Toluene); (e) Section: T3/T6/T9 (Benzene); (f) Section: T3/T6/T9 (Toluene).

be due to biodegradation and/or sorption to soil particles. The last two samplings reveal a situation very similar to the one on the 7th day after contamination, that is, high concentrations upstream, close to the entrance of water flow, and lower concentrations downstream, below the ones at the beginning of the experiment. These data suggest that 70 days after contamination B and T amounts distribution in water along the soil block is similar to the one at the beginning of the experiment, strongly suggesting microbial degradation in the central part of the block.

Higher amounts seen upstream at the end of the experiment could also be due to a saturation of soil particles which could no longer retain the contaminants, and would

then remain dissolved in water. Or, to the onset of anaerobic conditions in the soil block in the area close to the contamination source; despite the constant arrival of dissolved oxygen in the water flow, the increased biodegrading activity could be depleting the environment of oxygen in a faster rate than its arrival with the water flow. Much lower amounts seen downstream on the 84th day are most probably due to the action of biodegrading soil microorganisms as well.

### 3.2. Soil analysis

For chemical and microbiological analysis, three soil samples, A, B, and C (Fig. 2), were collected 0.30 m,

0.90 m and 1.20 m, respectively, from the upstream reservoir, 85 days after contamination, 0.50 m below the soil block surface.

Soil chemical analysis evidenced higher amounts of benzene than of toluene, as shown in Table 6. Data from statistical analysis of soil samples also showed that B and T amounts are significantly different at each sampled location ( $p = 0.0574$ ). Benzene seems to present higher affinity for the tested soil than toluene, and/or higher degradability, as its amounts decrease significantly in downstream samples, attaining the same levels as toluene. The sampled points are also significantly different among them concerning contaminants amounts ( $p = 0.197$ ).

The retention of benzene and toluene by soil enables the interaction between bacteria and these hydrocarbons. However, higher retained concentrations may indicate greater difficulty of the compound to be degraded.

Results presented in Table 7 show microbial activity existing in the contaminated soil.

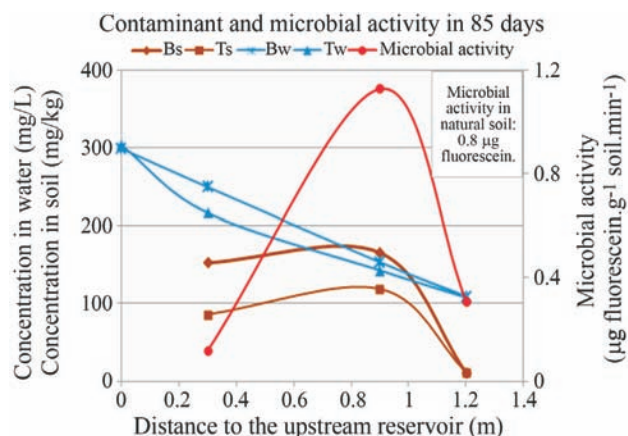
Figure 16 shows microbial activity and amounts of B and T in soil and water samples on the 83<sup>rd</sup> day after contamination. Microbial activity is still low at the entrance of the block (sample A), even after 2.5 months of contaminant presence in soil. It would be expected that the microbiota would have adapted by then and the degrading activity be higher. This low activity could be due to a decrease in biomass caused by water flow carrying microbial cells downstream, as mentioned previously. The double impact on the microbiota, first the shift from unsaturated to saturated environment, followed by the entrance of contaminated water, could have delayed their adaptation which could still not be completed after 85 days, explaining the low activity. Also, biodegradation may have already taken place and, despite the constant input of dissolved oxygen with the water flow, redox potentials near the upstream reservoir may already be low, tending to the anaerobic degradation of com-

**Table 6** - Results of the chromatography analysis of benzene and toluene in the soil.

| Samples | Benzene<br>(mg kg <sup>-1</sup> ) | Standard<br>deviation | Toluene<br>(mg kg <sup>-1</sup> ) | Standard<br>deviation |
|---------|-----------------------------------|-----------------------|-----------------------------------|-----------------------|
| A       | 153.00                            | 67.29                 | 86.33                             | 26.41                 |
| B       | 166.33                            | 48.64                 | 119.00                            | 35.17                 |
| C       | 11.67                             | 4.93                  | 12.00                             | 8.19                  |

**Table 7** - Microbial activity of the contaminated soil.

| Samples | $\mu\text{g fluorescein} \times \text{g}^{-1} \text{ soil} \times \text{min}^{-1}$ | Standard deviation |
|---------|------------------------------------------------------------------------------------|--------------------|
| A       | 0.12                                                                               | 0.00               |
| B       | 1.13                                                                               | 0.16               |
| C       | 0.31                                                                               | 0.08               |



**Figure 16** - Microbial activity in soil, B and T amounts in soil ( $B_s$  and  $T_s$ ) and water samples ( $B_w$  and  $T_w$ ) on the 85<sup>th</sup> day of experimentation.

pounds while aerobic activity decreased. Biodegradation may have become faster than oxygen input. Besides, determinations of microbial total activity were performed under aerobic conditions and did not measure anaerobic activity. Other studies and analyses would be necessary to better understand and explain these results. Downstream (sample B), microbial activity increases to higher levels than those measured in the natural soil, to then decrease next to the downstream reservoir (sample C). Concomitantly, contaminants amounts in water decrease along the soil block, while those retained in soil samples remain unchanged until the second half of the block, decreasing next to the downstream end. This decrease in B and T concentrations in soil follows the increase in microbial activity. On the other hand, the enhanced microbial activity does seem to affect the rate of contaminants removal from water. These data suggest that biodegradation happens mostly for contaminants adsorbed on soil, while a lesser decrease is seen in water samples. As B and T are transported dissolved in water along the soil block, and degraded, what is retained in the soil matrix is further degraded by soil microbiota, which tends to remain preferentially on soil particles and aggregates rather than in suspension (Ranjard & Richaume, 2001). Differences in B and T amounts seen in soil samples but not in water also corroborate this possibility, because of different biodegradability of both compounds. Thus, a constant small decrease is observed in contaminants concentrations in water, while in soil their amounts seem related to microbial activity.

Sample C shows a very reduced microbial activity, corresponding to almost undetected amounts of contaminants by the methodology adopted. The enhanced biodegrading activity is not maintained as soil contamination decreases.

In soil, there is a significant difference in B and T amounts between the sampling points. Thus, statistical analysis corroborates the conclusion that degradation is

probably more accentuated on soil particles and/or inside soil aggregates than in water, and that T concentrations are generally lower in soil samples.

#### 4. Conclusions

The paper presented results obtained from an *in-situ* experiment where water contaminated with both benzene and toluene was allowed to flow through an isolated block composed of residual soil of gneissic origin. A number of conclusions could be drawn regarding both the assembly and validity of the experiment as well as biodegradation properties of both organic compounds inside the residual soil mass.

Regarding the assembly of the experiment, it appears that the methodology used in the sample preparation proved to be satisfactory. A number of experimental issues had to be addressed as for example block excavation/isolation and proper sealing of its faces, installation of monitoring tubes for water samples collection, maintenance along time of hydraulic heads at block boundaries and contaminant concentrations in the upstream reservoir. The obtained results indicate that these issues were properly addressed.

Chemical and microbiological analyses showed decay in concentrations of benzene and toluene in water with time. Both compounds presented similar decay profiles in water, indicating no significant differences in retention and biodegradation of B and T under the conditions of this study.

One single sampling of soil, performed at the end of the experiment, showed a higher concentration of benzene than of toluene in the upstream region of the block, possibly because of its higher retention and/or lower degradation. Both benzene and toluene amounts decreased to the same level in the downstream region, indicating higher degradation at the center portion of the block, where a peak in microbial activity was also measured in the same sampling. These results suggest that biodegradation of these organic compounds may happen preferentially on soil particles and/or in soil aggregates.

Levels of aerobic activity along the block coupled with contaminants concentrations throughout the experiment suggest the onset of anaerobic degradation upstream during the final weeks of the monitoring period.

#### Acknowledgments

The authors would like to acknowledge the National Council for Scientific and Technological Development (CNPq), the Support Research Foundation of the State of Minas Gerais (FAPEMIG) and the Coordination of Superior Level Personnel Improvement (CAPES) for the financial support.

The authors thank Amanda F.B. Fernando for precious help with soil chemical and microbiological analyses.

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# Development of an Instrumented Channel for Multiphase Flow in Unsaturated Soils

R.P. Sousa, I.B. Oliveira, S.L. Machado, E.A. Sales

**Abstract.** This paper presents the development of an instrumented channel for two-dimensional multiphase flow of automotive fuels in unsaturated soils. Flow experiments were performed using water or diesel in an eolian sand at residual water saturation, a particular case of multiphase flow. Aspects such as the visual monitoring of the infiltration experiments and the great reproducibility of the developed experimental protocol can be cited as highlights of this research. The two sets of tensiometers, hydrophilic and hydrophobic, used during the diesel experiment, responded differently. While the hydrophobic tensiometer, filled with diesel, measured the full range of diesel suctions, the hydrophilic tensiometers, filled with water, responded less effectively to the passage of the diesel wetting front. Flow experiments were modeled using Philip's equation, considering the flow in unsaturated conditions above the wetting front. The time required for the diesel wetting front to reach the tensiometers was about 8.8 times higher than for water, considering the tensiometer readings, and, about 6.1 times higher than for water considering the visual observations. These values are comparable to the ratio of fluid mobilities  $[(\rho_w/\rho_w)/(\rho_{diesel}/\rho_{diesel}) = 5.5]$  of water and diesel.

**Keywords:** physical modeling, unsaturated soils, multiphase flow.

## 1. Introduction

Since the seventies, many theoretical and experimental studies have been performed to describe the flow and solute transport of non-aqueous phase liquids (NAPL) through unsaturated soil, the capillary fringe and within the aquifer, as reviewed by Abriola (1989). Abriola & Pinder (1985) elaborated the first mathematical and computational code to model the flow of NAPL in the subsurface, a very complete multiphase and multi-component 3D model, considering mass transfer between phases. Kaluarachchi & Parker (1989) developed a multiphase 3D model for the flow through both saturated and unsaturated zones. Concerned about the predictive ability of the numerical models, Kueper *et al.* (1989) highlighted the need for experimental studies in order to understand the multiphase flow under various hydrogeological conditions, as the predictions have to be compared with data obtained in the laboratory or in the field.

The literature reports the development of many experimental devices used to generate data to model multiphase flow. For example, Silliman *et al.* (2002) presented the development of two-dimensional experiments on solute transport in the capillary fringe of homogeneous and heterogeneous porous media. McDowell & Powers (2003) analyzed the distribution of gasoline with 10% alcohol by volume in a two-dimensional flow study conducted in the vadose zone and capillary fringe. The authors used imag-

ing techniques, with emphasis on the partitioning of ethanol in the different soil phases. The experimental work of Kamon *et al.* (2004) characterized the flow of DNAPL in the saturated zone, both considering or not the groundwater flow. The works of Oostrom *et al.* (2007) and Kamaruddin *et al.* (2011) present a comprehensive review of many laboratory and numerical studies on the migration of hydrocarbons in the subsurface environment, with an emphasis on DNAPL and LNAPL infiltration and redistribution.

Other examples of laboratory experiments performed in flow channels made use of different techniques to measure the transport parameters (Schiegg, 1990; Host-Madsen & Jensen, 1992; Oliveira, 1995; Butts & Jensen, 1996; Schroth *et al.*, 1998; Kamon *et al.*, 2004; and Kechavarzi *et al.*, 2000, 2005, 2008). The techniques described in these works are invasive and non-invasive. The non-invasive techniques used for soil moisture content determination are: X-ray and gamma ray attenuation, visible light reflection and transmission through the soil, analysis of multi-spectral images, radar (GPR) and three-dimensional seismic methods. The invasive techniques used for suction measurements are: tensiometry and electrical conductivity. The various liquids used by the researchers were: water, ethylene glycol, 4-chlorotoluene, n-hexanol, mineral oil, hydrofluoreter, BTEX, gasoline, Soltrol 220® and Jet Fuel, in different types of soils.

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Submitted on January 20, 2012; Final Acceptance on January 21, 2013; Discussion open until April 30, 2013.

In Brazil, many researchers (Corseuil & Marins, 1997; Kaipper, 2003; Silveira, 2004; Schneider, 2005; Amorim Jr, 2007) have been developing experimental studies on soil and aquifer contamination by Brazilian automotive fuels. However, most of these studies are developed in the field in the southeast region of Brazil and they focus on NAPL transport after reaching the water table.

To generate specific data for multiphase flow of Brazilian automotive fuels in a variety of unsaturated soils, the present research developed a two-dimensional instrumented channel to carry out laboratory studies of NAPL transport in unsaturated soils. It is planned to test soils collected in the Metropolitan area of Salvador: sand dune (non reactive soil); regolith (sandy silty clay soil); tertiary sediments (clayey sand soil); and an expansive clay soil. This paper describes the development and testing of the experimental apparatus, using water and diesel as the permeating fluids, at residual water saturation, and sand dune as the porous medium. Future studies will address diesel permeation through the other three soil types, useful information for describing the Brazilian automotive fuel contamination in the studied tropical region, information not available in the literature.

## 2. Materials and Methods

### 2.1. Soil and liquid characteristics

The soil used in the experiments is a Quaternary sand dune from the Metropolitan Region of Salvador, state of Bahia, Brazil, with 100% of sand content, classification NBR 6502 - medium to fine sand and classification SUCS - SP poorly graded sand. The sand has a grain density of  $\rho_s = 2.68 \text{ g/cm}^3$  and an organic content given in total volatile solids of TVS = 0.091%. Grain size distribution and soil liquid retention curves are presented in Figs. 1 and 2, respectively.

For the soil water retention curves and the infiltration experiments in the flow channel, drinking water distributed by the state company EMBASA was used, at equilibrium with the laboratory temperature and atmospheric pressure, after losing its residual chlorine. The recommendation for using tap water in the infiltration experiments, instead of demineralized water, was given by Philip (1969), Bond & Collins-George (1981) and Klute (1986) to avoid chemical aggressiveness. For the soil diesel retention curves and the infiltration experiments in the

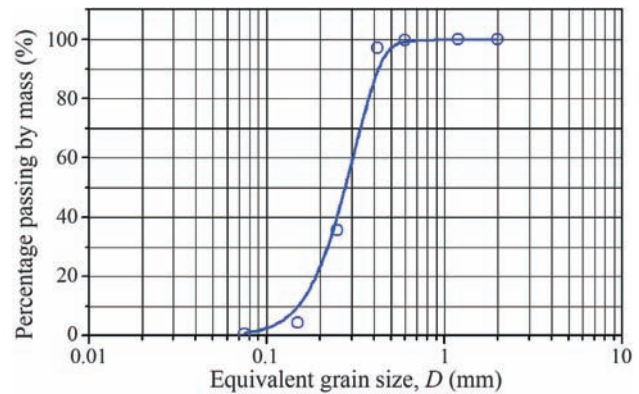


Figure 1 - Sand grain size distribution curve.

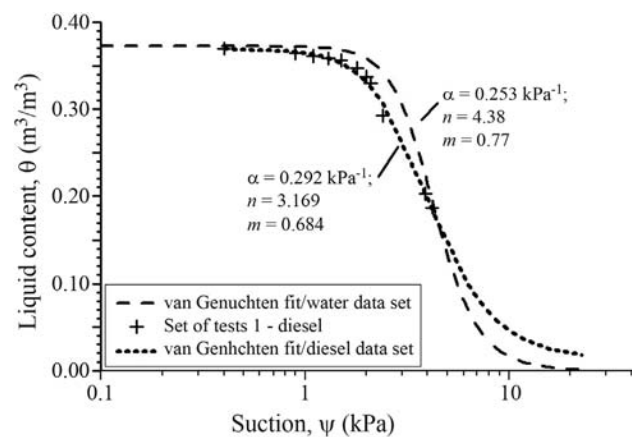


Figure 2 - Soil liquid retention curves (SLRC), drainage branch, using a vaporization chamber.

flow channel, commercial Brazilian diesel was used. From the literature, the properties of water and diesel at 29 °C, are given in Table 1.

The soil liquid retention curves (SLRC) shown in Fig. 2 were obtained with a vaporization camera described in Sousa *et al.* (2011), a procedure applied by many authors (Plagge *et al.*, 1990; Wendroth *et al.*, 1993; Tamari *et al.*, 1993; and Oliveira, 1995). To apply this method, the saturated soil with the liquid of interest is open to the atmosphere and subject to a gradual loss of liquid content. The suction measurements are made sequentially, using tensiometers, at the different stages of the drying process. Further information about this procedure can be obtained in

Table 1 - Liquid properties at 29 °C from Oliveira (2001).

| Liquids | Density<br>$\rho \text{ (g cm}^{-3}\text{)}$ | Viscosity<br>$\mu \text{ (cP)}$ | Mobility<br>$\rho/\mu \text{ (g cm}^{-3}\text{/cP)}$ | Dielectric<br>constant $\epsilon$ | Vapor pressure<br>PV (mmHg) | Surface tension<br>$\sigma \text{ (dina cm}^{-1}\text{)}$ |
|---------|----------------------------------------------|---------------------------------|------------------------------------------------------|-----------------------------------|-----------------------------|-----------------------------------------------------------|
| Water   | 0.9954                                       | 0.810                           | 1.229                                                | 80.08                             | 32.238                      | 71.38                                                     |
| Diesel  | 0.8323                                       | 3.75                            | 0.222                                                | 2.13                              | 2.46 E-4*                   | 26.78*                                                    |

\*Reference: Environmental Canada, ESTC (1997).

Sousa *et al.* (2011). The Van Genuchten (1980) model was used to fit the soil liquid retention curves as follows:

$$\Theta = \frac{1}{[1 + (\alpha\psi)^n]^m}, \quad (1)$$

$$\text{with } \Theta = \frac{\theta - \theta_r}{\theta_s - \theta_r}.$$

where  $\theta$ ,  $\theta_s$  and  $\theta_r$  [-] are the soil volumetric liquid content at a given suction  $\psi$  [kPa], at saturation and at residual state, respectively.  $\alpha$  [kPa<sup>-1</sup>],  $m$  [-] and  $n$  [-] are fitting parameters.  $m = 1 - 1/n$  was adopted. The fitting curve for the soil water retention curve (SWRC) was obtained combining three different sets of suction and water content data.

Table 2 presents some physical parameters for the sand dune used in the experiments and the fitting parameters for each individual SLRC.

## 2.2. Soil compaction protocol

To assure the homogeneity of the sand within the flow channel, a compaction protocol was developed. For this, a small chamber was built with dimensions and materials similar to those used for the flow channel. The chamber was made of 8 mm thick glass, with dimensions of 40 x 40 x 13.5 cm. The procedure from Oliveira *et al.* (1996) was used to compact the soil inside the chamber and the flow channel. For this, thin layers of air dried sand were spread inside the chamber, followed by pressuring each layer with a 4 kg metal compactor until it reached a thickness of about 4 mm and the desired bulk density:  $\rho = 1.72 \text{ g/cm}^3$ . In the case of the channel, each sand layer was uniformly distributed in the cross section and homogeneously pressured by a 7 kg metal compactor 270 times to achieve the same layer thickness and bulk density.

To verify the efficiency and reproducibility of the compaction protocol and to determine the soil capillary rise curve prior to the channel tests, capillary rise experiments were performed. In the compaction chamber, the water level was kept constant using two Mariotte tubes at each side. A layer of Geodrain (MacDrain 2L, Maccaferri of Brazil LTDA, Sao Paulo, SP) was used to uniformly distribute the water at the bottom. At the end of the experi-

ment, soil samples were collected along the vertical profile to determine the soil bulk density and the moisture content. This procedure enabled the determination of the wetting branch of the soil water retention curve.

## 2.3. Flow channel

The channel is made of stainless steel and glass, with nominal dimensions of 200 x 120 x 15 cm (Fig. 3).

A channel thickness of 15 cm was chosen to follow the recommendation of Schiegg (1990) to avoid the wall effect, using a maximum value for sand diameter of 2 mm. The channel sides and front walls are made of a 12 mm thick tempered glass to enable visual observation of the experiments. The channel extensions located on each side wall were built to hold two Mariotte tubes for water application at the channel bottom. To prevent plastic deformation of the channel front side, two steel bar lattices were used (Fig. 3). To avoid undesirable physico-chemical interactions between the organic liquids and the stainless steel and the consequent wall effects, the inner surfaces of the channel, the bottom and back walls were coated with epoxy and covered with a thin layer of sand, ensuring mineralogical similarity with the porous medium.

A total of 30 holes, with couplings made of aluminum, were made in the back wall of the channel to insert tensiometers for suction readings: 15 for water (A) and 15 for NAPL (G). During the soil compaction, the openings were kept closed with aluminum caps, and during the installation of the tensiometers the caps were carefully removed to avoid soil loss, mainly for sand dune, a cohesionless soil. The openings also allow soil sampling using small aluminum tubes with nominal sizes of 15 x 0.635 cm (internal diameter). The samples are used for liquid content measurement at the end of the experiment.

In order to allow water or NAPL infiltration at the soil surface, a stainless steel reservoir was built, with a porous bottom made of sintered bronze, nominal pore size of 5  $\mu\text{m}$  (Free Filters Industry and Commerce, Sao Paulo, SP, Brazil). A pressure transducer was installed inside the reservoir, just above the porous bottom, to monitor the liquid level during the experiments (Fig. 4).

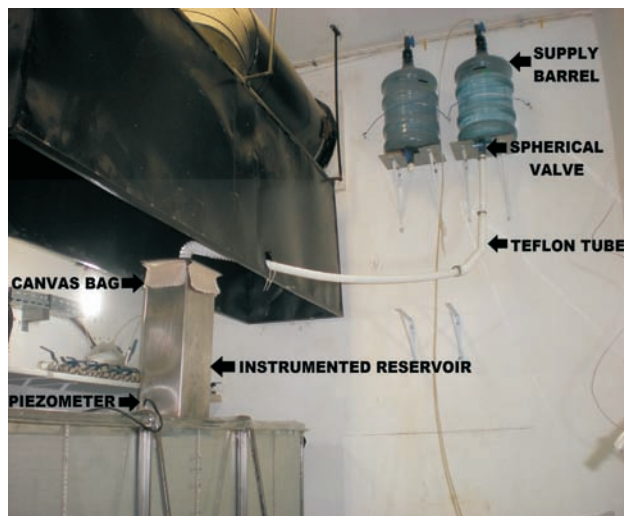
The reservoir has dimensions of 50 x 20 x 15 cm with a storage capacity of about 15 L. After preliminary tests of

**Table 2** - Physical parameters of sand dune obtained with vaporization cell and mathematical modeling.

| Experiment number | Permeability coefficient<br>$k \text{ (m s}^{-1}\text{)}$ | Bulk density<br>$(\text{kg.m}^{-3})$ | Soil liquid retention curve                |                                            |                                         |         |                               |
|-------------------|-----------------------------------------------------------|--------------------------------------|--------------------------------------------|--------------------------------------------|-----------------------------------------|---------|-------------------------------|
|                   |                                                           |                                      | $\theta_r$<br>$(\text{m}^3.\text{m}^{-3})$ | $\theta_s$<br>$(\text{m}^3.\text{m}^{-3})$ | Fitting parameters van Genuchten (1980) |         |                               |
|                   |                                                           |                                      |                                            |                                            | $m$ (-)                                 | $n$ (-) | $\alpha$ (kPa <sup>-1</sup> ) |
| #01 (water)       | $2.60 \times 10^{-4}$                                     | 1720                                 | 0.012                                      | 0.373                                      | 0.822                                   | 5.59    | 0.278                         |
| #02 (water)       | $2.60 \times 10^{-4}$                                     | 1724                                 | 0.015                                      | 0.371                                      | 0.803                                   | 5.08    | 0.222                         |
| #03 (water)       | $2.60 \times 10^{-4}$                                     | 1721                                 | 0.004                                      | 0.372                                      | 0.806                                   | 5.15    | 0.258                         |
| # 04 (diesel)     | $5.40 \times 10^{-5}$                                     | 1720                                 | 0.012                                      | 0.369                                      | 0.684                                   | 3.169   | 0.292                         |



**Figure 3** - View of the flow channel with sand compaction in progress. Features: steel bar lattice to reinforce the channel structure; back wall coated with epoxy & sand; sidewall reservoir to hold the Mariotte tube.



**Figure 4** - Instrumented liquid reservoir on the soil surface connected to a fluid supply barrel.

the liquid reservoir performance with water, it was concluded that to maintain the water flow rate compatible with the sand infiltration rate it was necessary to use a canvas bag with column format inside the reservoir. For the diesel, a more viscous fluid, the canvas bag proved inadequate, and a plastic bag in column format was used. At the begin-

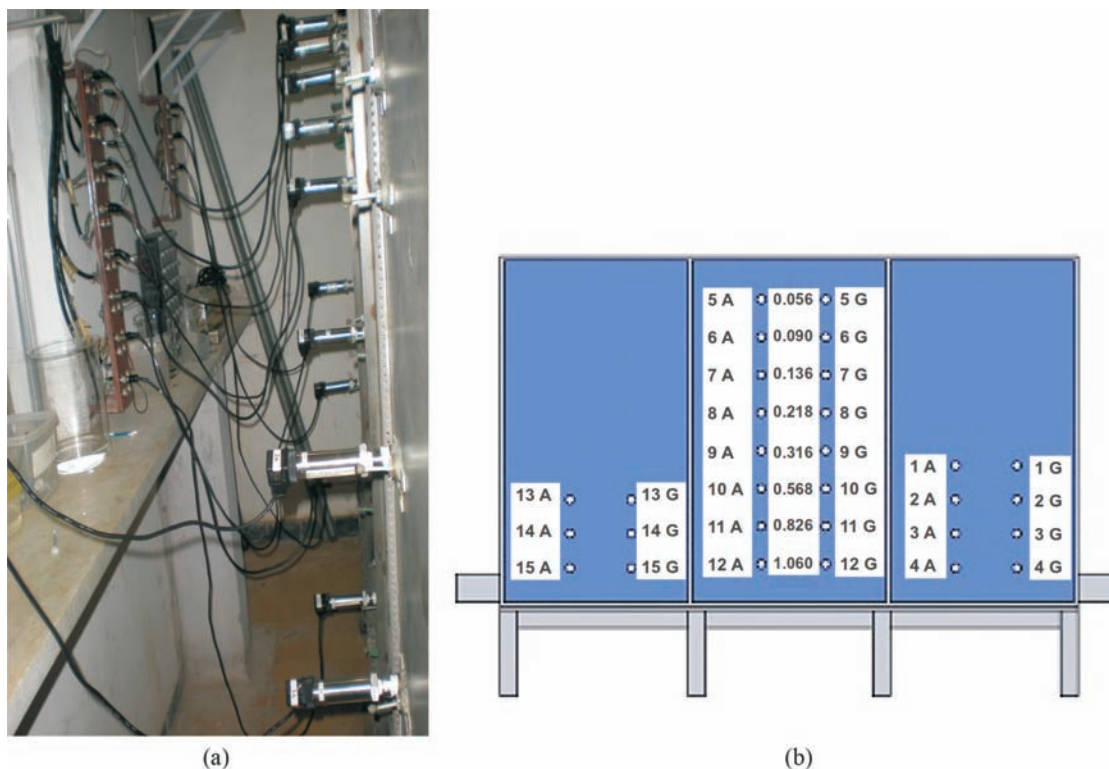
ning of each infiltration experiment, a supply barrel, connected with the reservoir, dispenses the liquid through a Teflon tube with diameter of 29 mm (Fig. 4).

To measure interstitial pressures, hydrophilic and hydrophobic tensiometers were used. A detailed description of the tensiometer; the tensiometer calibration process; and the hydrophobic surface treatment for NAPL suction are presented in Sousa *et al.* (2011). For the infiltration experiments with water, hydrophilic tensiometers (filled with water) were used, whereas for diesel infiltration experiments both hydrophilic (filled with water) and hydrophobic tensiometers (filled with diesel) were used. The tensiometers consist of a pressure transducer with suction range from 0 to -100 kPa, connected to a stainless steel sleeve, with a porous tip made of alumina ceramic material (Model-0604D04 B01M1, Soilmoisture Equipment Corporation, USA). To saturate the tensiometers and its porous tips with the liquid of interest, a cylindrical vacuum chamber with nominal dimensions of 30 x 19.9 cm (internal diameter) was built in acrylic and aluminum. A high flow rate vacuum pump (Model D16A, Leybold Heraeus, 1 hp, Germany) extracts the air from the chamber. Figure 5 shows the 15 tensiometers inserted in the back wall of the channel to monitor the water infiltration test. To prevent the porous tips of the tensiometers from drying out, they must be inserted just before the test. A computer system (PLC Controller and Model-MCI02 QC, HI Technology Industry and Trade Ltda., Sao Paulo, SP, Brazil) for data acquisition and storage is connected to the tensiometers and the pressure transducer is located at the bottom of the liquid reservoir. Although not shown in this paper, the experimental apparatus has another set of tensiometers which are able to measure suction values of up to 1000 kPa (Epron EPX-NO1, Entran Sensors & Eletronics, Fairfield, NJ, USA), as described by Barreto *et al.* (2007). This set of tensiometers may be used in tests involving clayey soils.

## 2.4. Infiltration experiments and uni-dimensional modeling

Six infiltration experiments were performed, five with water and one with diesel. Table 3 shows the conditions observed for each of the six flow channel experiments. An average bulk density of  $\rho = 1.714 \text{ g/cm}^3$  was achieved for the six experiments, which is very close to the bulk density obtained in the compaction chamber ( $\rho = 1.72 \text{ g/cm}^3$ ), attesting the good reproducibility of the compaction protocol. At the beginning of each infiltration experiment, the supply barrel dispensed the liquid within the reservoir in about 25 s. Water/diesel interstitial pressures and the liquid level inside the reservoir were recorded every 2 s. The visual monitoring of the infiltration experiments was drawn by hand, tracing the successive wetting front positions on the channel front wall.

After finishing the experiments, soil samples were collected to determine the soil liquid content. The soil suc-



**Figure 5** - (a) Channel back wall with attached tensiometers connected to the data acquisition system and (b) schematic view of the positions of the tensiometers. Numbers indicate the depth of the installed tensiometers (m).

tion values were recorded just before the removal of the tensiometers from the wall in order to determine a soil liquid retention curve (scanning curve). The water content was determined gravimetrically following the standard procedures. The diesel content was also determined gravimetrically, after keeping the samples in a muffle for 2 h at 600 °C (see Sousa *et al.*, 2011). The initial water and organic contents of the samples were discounted in order to compute the diesel content (the soil water content was assumed constant during the tests with diesel).

To simulate the advance of the wetting front for water and diesel, the infiltration model proposed by Philip (1969) was used, as follows:

$$I(t) = L(T)(\theta_s - \theta_i) = S t^{1/2} + \frac{2}{3} k t \quad (2)$$

where  $I(t)$  and  $L(t)$  [L] are respectively the water infiltration and the wetting front position;  $k$  [ $\text{LT}^{-1}$ ] is the coefficient of permeability;  $h_p$  and  $h_f$  [L] are the hydrostatic head at the soil surface and the suction head at the wetting front respectively;  $\theta_i$  [-] is initial soil volumetric water content and  $S$  [ $\text{LT}^{-1/2}$ ] is the soil sorptivity defined by Philip (1969) as follows:

$$S = \sqrt{2 k (h_p - h_f) (\theta_s - \theta_i)} \quad (3)$$

**Table 3** - Some physical parameters related to the five flow channel experiments.

| Experiment number | Soil height (cm) | Soil mass (kg) | Averaged bulk density ( $\text{g cm}^{-3}$ ) | Volume of liquid infiltrated ( $\text{cm}^3$ ) | Elapsed time to reach capillary fringe (min) |
|-------------------|------------------|----------------|----------------------------------------------|------------------------------------------------|----------------------------------------------|
| #01- water        | 115              | 636.9          | 1.720                                        | 8813.0                                         | 25.3                                         |
| #02- water        | 115              | 657.7          | 1.690                                        | 8205.2                                         | 30.6                                         |
| #03- water        | 115              | 624.4          | 1.721                                        | 9420.8                                         | 31.1                                         |
| #04- water        | 115              | 625.8          | 1.720                                        | 10940.2                                        | 31.1                                         |
| #05- water        | 115              | 626.4          | 1.719                                        | 9420.8                                         | 29.4                                         |
| #06- diesel       | 115              | 624.8          | 1.712                                        | 14524.5                                        | 214.0                                        |

The value of  $\theta_s$  was assumed as the average soil porosity and  $\theta_i$  was the average water content of the soil prior test beginning. Values of  $h_p$  were measured during the infiltration test by the pressure transducer installed at the bottom of the liquid reservoir.  $h_f$  was assumed as the average value of the initial tensiometer readings. The value of  $k$  was assumed as the average soil coefficient of permeability.

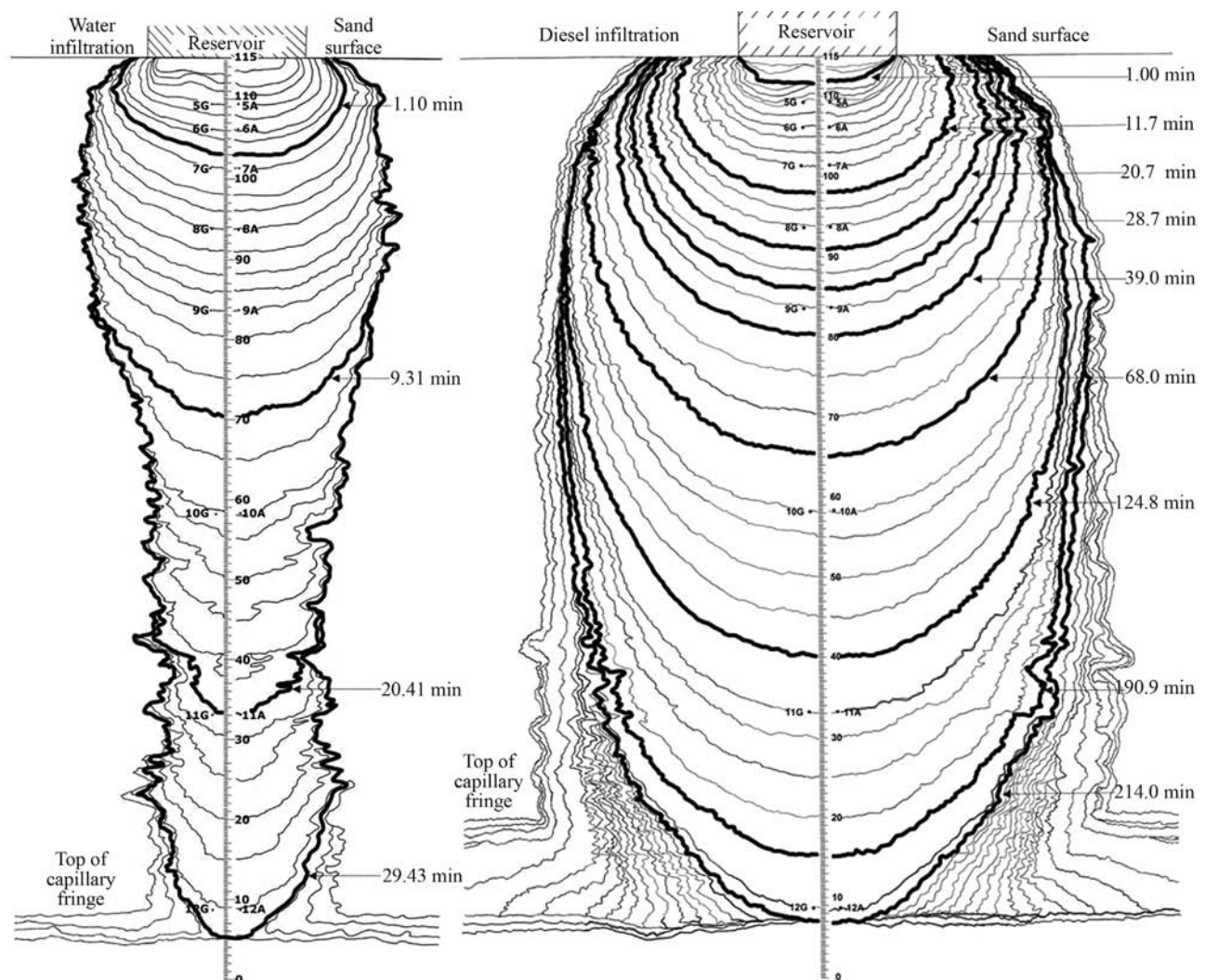
### 3. Results and Discussion

Figure 6 shows the wetting front advance for both water (experiment #05) and diesel (experiment #06). The lines were drawn on the front glass wall of the channel during the visual observation of the flow experiment. Isochrones approximately each 10 min are highlighted.

Considering the visual observations, the time for diesel wetting front to reach the capillary fringe was about 7.3 times longer than for water (214.0 min/29.43 min), a value slightly superior to the mobility ratio water/diesel [ $(\rho_w/\rho_w)/(\rho_{diesel}/\rho_{diesel}) = 5.5$ ].

Table 4 presents the time for the wetting front to reach the tensiometers from the tensiometer readings (TR) and from visual observation in the front face of the channel (VO).

It can be seen in Table 4 that for smaller depths, during the water infiltration experiments, the tensiometer detected the water front passage earlier than the visual observations, while the opposite occurred during the diesel experiment. This had been expected from the outset of the experiments. First, the water tensiometers responded quickly (1 to 3 s) to a change in the water pressure; and secondly, the water wetting front progresses so fast at the beginning of the infiltration that it was more difficult to draw the first two isochrones on the channel front wall. For the diesel, the wetting front progresses considerably slower than water, enabling better visual monitoring through the channel front wall, and the response time for the diesel tensiometers was about 35 s.



**Figure 6** - Visual observation of the wetting front advance a) water experiment and b) diesel experiment.

**Table 4** - Results for the time measurement from visual observations (VO) and that from tensiometer readings (TR).

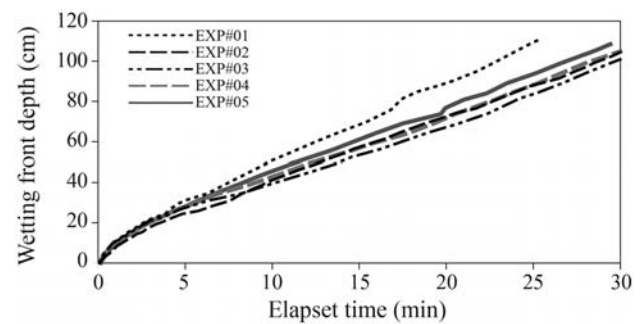
| Depth<br>(m) | EXP#01 - Water |               |              | EXP#02 - Water |               |              | EXP#03 - Water |               |              | EXP#04 - Water |               |              | EXP#05 - Water |               |              | EXP - Diesel oil |               |              |
|--------------|----------------|---------------|--------------|----------------|---------------|--------------|----------------|---------------|--------------|----------------|---------------|--------------|----------------|---------------|--------------|------------------|---------------|--------------|
|              | Elapsed time   |               |              | Elapsed time   |               |              | Elapsed time   |               |              | Elapsed time   |               |              | Elapsed time   |               |              | Elapsed time     |               |              |
|              | V.O.<br>(min.) | T.R.<br>(min) | Diff.<br>(%) | V.O.<br>(min.) | T.R.<br>(min) | Diff.<br>(%) | V.O.<br>(min.) | T.R.<br>(min) | Diff.<br>(%) | V.O.<br>(min.) | T.R.<br>(min) | Diff.<br>(%) | V.O.<br>(min.) | T.R.<br>(min) | Diff.<br>(%) | V.O.<br>(min.)   | T.R.<br>(min) | Diff.<br>(%) |
| 0.056        | 0.28           | 0.23          | 15           | 0.60           | 0.57          | 6            | 0.29           | 0.18          | 38           | 0.24           | 0.27          | -10          | 0.38           | 0.22          | 42           | 2.39             | 4.03          | -69          |
| 0.090        | 0.61           | 0.42          | 32           | 1.00           | 0.68          | 32           | 0.62           | 0.25          | 60           | 0.65           | 0.43          | 34           | 0.71           | 0.38          | 46           | 3.96             | 5.67          | -43          |
| 0.136        | 1.29           | 1.07          | 17           | 1.90           | 1.65          | 13           | 1.38           | 0.98          | 29           | 1.40           | 1.20          | 14           | 1.40           | 1.22          | 13           | 7.96             | 9.57          | -20          |
| 0.218        | 2.74           | 2.68          | 2            | 3.82           | 3.40          | 11           | 2.93           | 2.42          | 18           | 2.69           | 2.87          | -6           | 2.86           | 2.72          | 5            | 17.02            | 18.37         | -8           |
| 0.316        | 4.88           | 6.45          | -32          | 7.50           | 6.47          | 14           | 6.23           | 4.72          | 24           | 5.67           | 4.50          | 21           | 5.54           | 5.08          | 8            | 32.72            | 34.37         | -5           |
| 0.568        | 11.44          | 17.58         | -54          | 14.56          | 15.93         | -9           | 16.13          | 12.85         | 20           | 14.55          | 12.90         | 11           | 13.35          | 11.88         | 11           | 83.77            | 86.97         | -4           |
| 0.820        | 17.44          | -             | -            | 22.99          | 25.45         | -11          | 24.14          | 21.05         | 13           | 22.71          | 20.60         | 9            | 20.91          | 19.58         | 6            | 142.9            | -             | -            |
| 1.062        | 24.08          | -             | -            | 30.37          | 28.45         | 6            | 0.00           | 29.18         | -            | 30.02          | 28.87         | 4            | 28.45          | 27.15         | 5            | 209.14           | 232.07        | -11          |

Notes: Elapsed time: time required for the wetting front to reach the tensiometer depth. Diff.: Difference between the visual observation and the tensiometer readings.

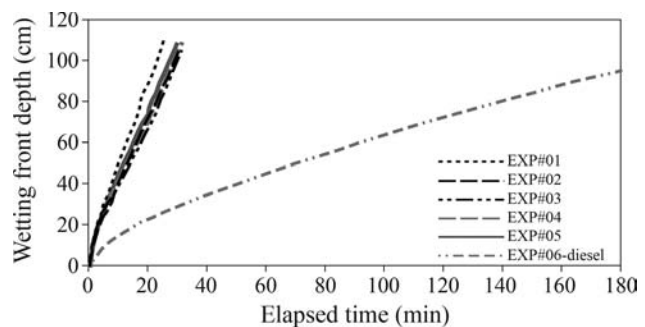
Figure 7 shows the progress of the wetting front position for the five water infiltration experiments. They are very close to each other (except test#01), attesting the reproducibility of the experiment. Figure 8 compares the results obtained for water and diesel, considering the wetting front from visual observations.

Figure 9 compares the readings of the tensiometers 5A, 7A and 8A for the five water infiltration experiments. Figure 9 shows that the tensiometers readings present good reproducibility. Although the initial suction measured by each tensiometer can be different, all tensiometers recorded the wetting front passage similarly. It also can be seen that 1 to 2.5 min after the passage of the wetting front, the suction increases gradually, indicating a drainage phenomenon. This is physically consistent with the fact that this is a variable head experiment and that infiltration velocities, lower than soil saturated permeability (see Fig. 12), will require some value of suction in the soil hydraulic function to equalize the infiltration rate.

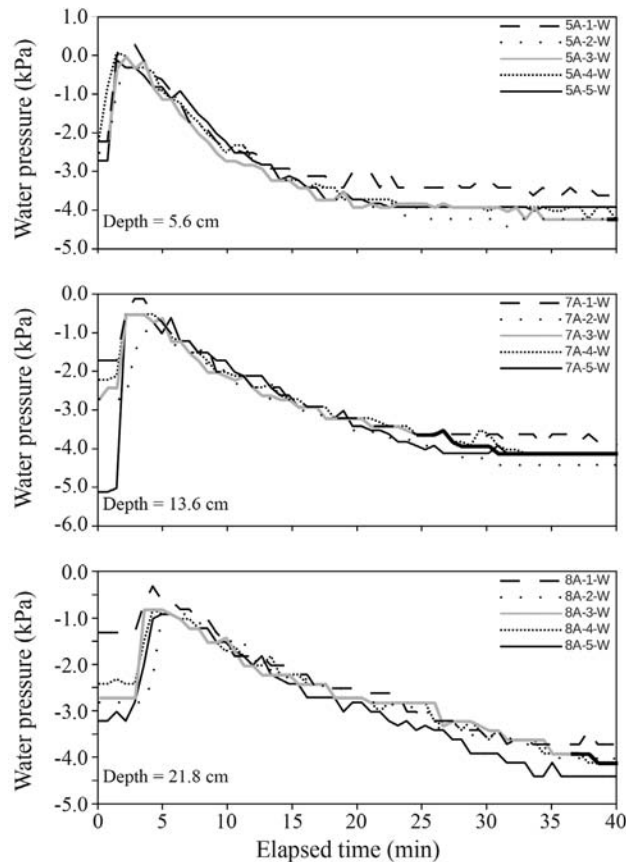
Figure 10 shows the tensiometer readings (5G, 7G and 8G) for the diesel experiment. The arrows indicate the beginning of the drainage process. It can be seen that the tensiometer 8G did not become fully saturated, making it difficult to detect the soil drainage process. A comparison between Figs. 9 and 10 shows that for the diesel experiment the soil remained saturated for a longer period of time and the drainage process was much less pronounced.



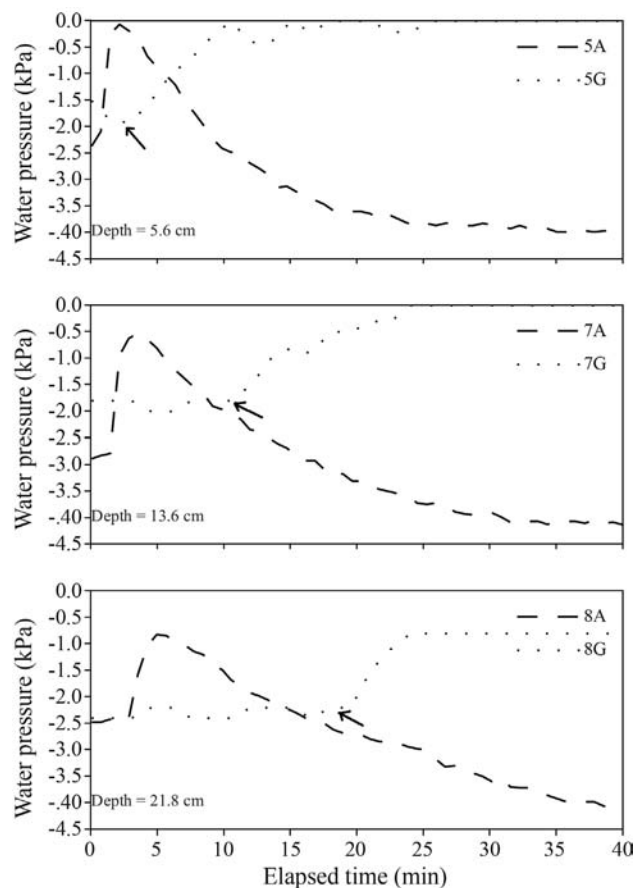
**Figure 7** - Position of the water wetting fronts as a function of time.



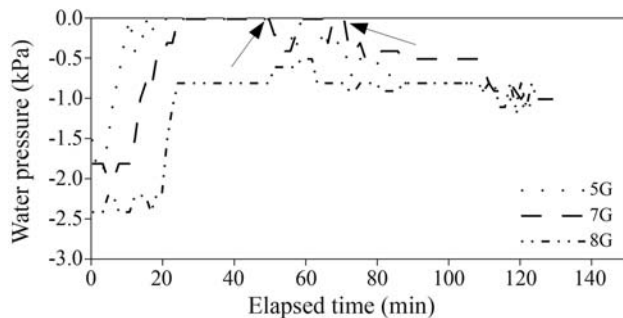
**Figure 8** - Water and diesel wetting fronts position as a function of time (Visual observation).



**Figure 9** - Tensiometer readings in the performed water infiltration tests.



**Figure 11** - Tensiometer readings in the water (average values) and diesel infiltration tests.



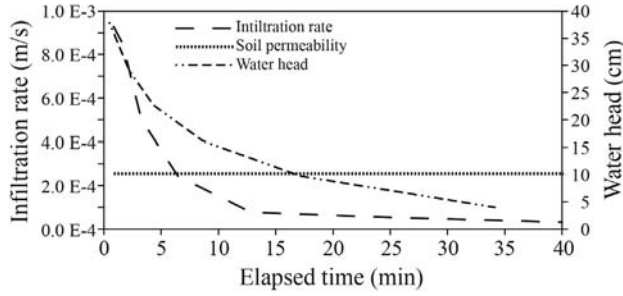
**Figure 10** - Tensiometer readings in the diesel infiltration test.

Figure 11 compares the results of the average water interstitial pressure measurements in tensiometers 5, 7 and 8 (line A) to the obtained values in the diesel experiment (line G). The arrows in Fig. 11 indicate the passage of the wetting front detected by the diesel tensiometers. The ratios between the time required for the diesel wetting front to reach a tensiometer to that for water were 13.7 (tensiometer 5), 7.8 (tensiometer 7) and 6.5 (tensiometer 8) with an average value of 8.8. Greater ratios occurred for the shallower depths (also see Table 4, TR column). Considering the visual observations, an average value of 6.1 is obtained.

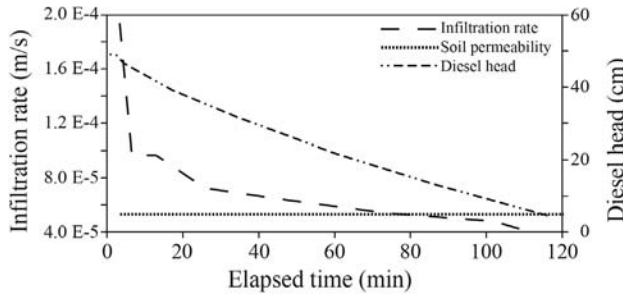
These results are comparable with the mobility ratio  $[(\rho_w/\rho_w)/(\rho_{diesel}/\rho_{diesel}) = 5.5]$  of liquids, although they are slightly higher than that (see Table 1). The most probable explanation for this discrepancy is illustrated in Fig. 6. As can be observed, the spreading of the wetting front in the case of the diesel experiment is higher than that for water. This requires a higher infiltration volume on the soil surface to achieve the same depth below the surface, justifying why the time required for the diesel wetting front to reach the tensiometers was higher than expected.

Figure 12 shows the average infiltration rate and the average variation in the water head inside the reservoir as a function of time for the five water experiments. As can be observed, after 6.4 min the average infiltration rate goes below the soil saturated coefficient of permeability ( $k_w = 2.60 \times 10^{-4}$  m/s). Figure 13 shows the same results as Fig. 12, now considering the diesel experiment. After only 75 min the infiltration rate goes below the soil saturated coefficient of permeability ( $k_{diesel} = 5.40 \times 10^{-5}$  m/s).

The behavior observed in Fig. 12 for water is compatible with the drainage process illustrated in Fig. 9. The results presented in Fig. 13 indicate that the drainage process



**Figure 12** - Average values of infiltration rate and water head inside the reservoir for water infiltration tests.



**Figure 13** - Average values of infiltration rate and diesel head inside the reservoir for diesel infiltration tests.

in the diesel experiment is much less pronounced, which agrees with what is illustrated in Fig. 10.

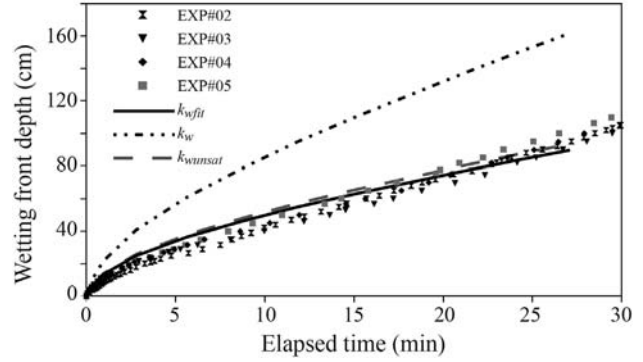
The results presented in Figs. 7 and 8 were modeled with Philip's (1969) infiltration equation. In order to take into account the variations in the liquid head inside the reservoir ( $h_p$ ) during the experiment, Eq. 2 was rewritten in an incremental way:

$$\Delta L = \frac{\frac{\partial L(t, h_p)}{\partial t} \Delta t + \frac{\partial L(t, h_p)}{\partial h_p} \Delta h_p}{\theta_s - \theta_i} \quad (4)$$

or

$$\Delta L = \frac{\left(0.5 S t^{-\frac{1}{2}} + \frac{2}{3} k\right) \Delta t + \left(0.5 \sqrt{2 k(\theta_s - \theta_i) t (h_p - h_f)^{-\frac{1}{2}}}\right) \Delta h_p}{\theta_s - \theta_i} \quad (5)$$

Figure 14 presents the modeling results for the water infiltration tests (Exp#2, #3, #4 and #5). Average values of  $\theta_s = 0.37$  and  $\theta_i = 0.001$  were used for both liquids and the average value of  $h_f = -27$  cm was adopted for water. The adopted values for  $h_p$  were those shown in Fig. 12. As can be observed, the use of the value of  $k_w = 2.60 \times 10^{-4}$  m/s obtained in laboratory (Table 2) leads to an overestimation of the wetting front depth. This is probably due to the fact that in the case of the Philip's equation a piston flow scenario is considered. In this scenario the infiltration rate is always higher than the saturated permeability of the soil and there is no drainage process, contrary to what was observed experimentally in this paper. In order to take into account the



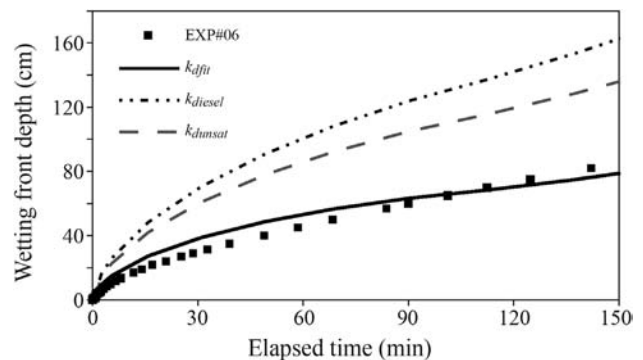
**Figure 14** - Experimental results for water and curves modeled with Eq. 6.

occurrence of the drainage process in the performed tests a new value of  $k$  was adopted ( $k_{wunsat}$ ) now considering the average value of soil suction during the drainage process. The Eqs 1 and 6 were used to compute a value of  $k_{wunsat} = 1.21 \times 10^{-4}$  m/s, corresponding to a soil suction of 2.88 kPa.

$$k(\Theta) = k_{sat} \Theta^{\frac{1}{2}} \left[ 1 - \left( 1 - \Theta^{\frac{1}{m}} \right)^m \right]^2 \quad (6)$$

Figure 14 also presents the results of the model for the best fitting value of  $k$  ( $k_{fit} = 1.14 \times 10^{-4}$  m/s,  $R^2 = 0.967$ ). This value is very close to the value of  $k_{wunsat}$ , which reflects the unsaturated conditions developed above the wetting front for all the water infiltration experiments.

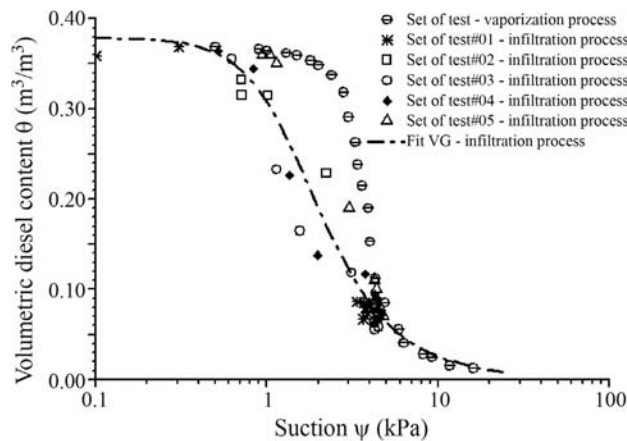
Figure 15 presents the modeling results for the diesel infiltration test. An average value of  $h_f = -21.6$  cm was adopted for diesel. The adopted values for  $h_p$  are shown in Fig. 13. As in the case of the water tests the value of  $k_{diesel} = 5.40 \times 10^{-5}$  obtained in laboratory (Table 2) leads to an overestimation of the wetting front depth. A value of  $k_{dunsat} = 4.27 \times 10^{-5}$  m/s was obtained considering an average value of suction of 1.16 kPa. In this case, the value of  $k_{dunsat}$  is closer to the value of  $k_{diesel}$  which is coherent with the fact that in the case of the diesel experiment the drainage process was much less evident. The wetting front advance is still overestimated in this



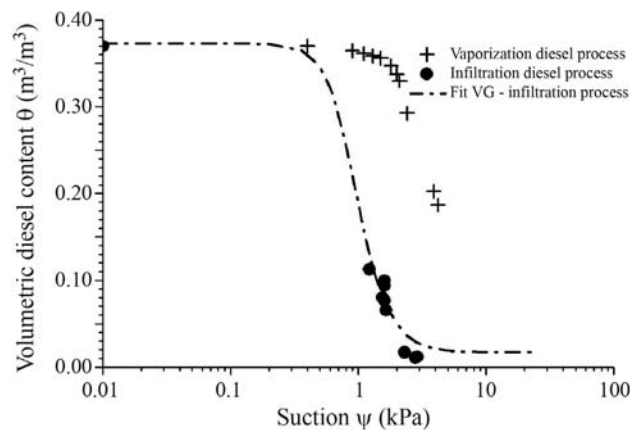
**Figure 15** - Experimental and predicted results for diesel.

case even considering the value of  $k_{dmsat}$ . Again, the most probable explanation for this discrepancy is higher lateral spreading of the wetting front in the case of the diesel experiment. The best fitting of the experimental results yields a value of  $k_{dfr} = 2.03 \times 10^{-5}$  m/s ( $R^2 = 0.956$ ).

The values of the liquid content and suction measured at the end of the infiltration experiments were used to determine soil liquid retention curves for water and diesel. Ta-



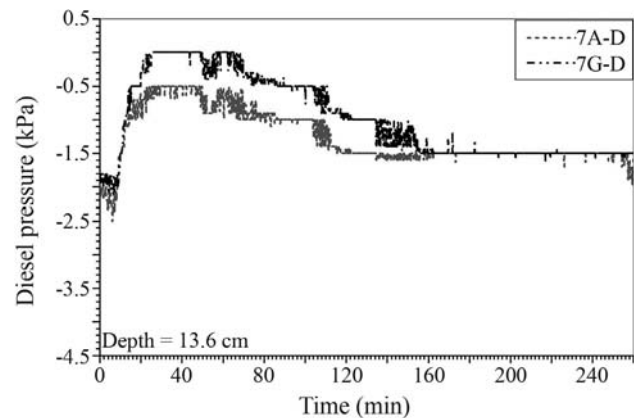
**Figure 16** - Soil liquid retention curves for water.



**Figure 17** - Soil liquid retention curves for diesel.

ble 5 summarizes the fitting parameters obtained. Figures 16 and 17 compare the soil liquid retention curves obtained at the end of the experiments with the main drainage retention curves presented in Fig. 2. As expected, the new curves are located to the left of the main drainage curve. However, the departure from the main drainage curve in Fig. 16 is much less remarkable than for the soil diesel retention curve (Fig. 17). This behavior agrees with the observations made above, based on Figs. 9 and 10, that in the water experiments the drainage process of the soil began earlier and was more evident than in diesel tests.

Figure 18 shows the results obtained when comparing the water and diesel tensiometer readings (7A and 7G) in the diesel experiment. As said above, the “A” tensiometers have a standard ceramic tip (hydrophilic) and are filled with water whereas the “G” tensiometers have a silanized ceramic tip (hydrophobic) and are filled with diesel. As can be observed, tensiometers “A” and “G” responded differently. While the hydrophobic tensiometer measured the full range of diesel suctions, the hydrophilic tensiometers responded less effectively to the diesel wetting front passage, showing the importance of using the hydrophobic tensiometer in the diesel experiments.



**Figure 18** - Readings of the tensiometers 7A and 7G during diesel infiltration test.

**Table 5** - Fitting parameters of the soil liquid retention curve using van Genuchten (1980) model.

| Equipment         | Experiment number                     | $\alpha$ (kPa <sup>-1</sup> ) | $m$    | $n$    | $\theta_s$ (m <sup>3</sup> .m <sup>-3</sup> ) | $\theta_r$ (m <sup>3</sup> .m <sup>-3</sup> ) | $R^2$  |
|-------------------|---------------------------------------|-------------------------------|--------|--------|-----------------------------------------------|-----------------------------------------------|--------|
| Water experiments | #01                                   | 0.7984                        | 0.5850 | 2.4120 | 0.3730                                        | 0.001                                         | 0.9870 |
|                   | #02                                   | 0.6813                        | 0.6180 | 2.3580 | 0.3840                                        | 0.001                                         | 0.9612 |
|                   | #03                                   | 0.9782                        | 0.5468 | 2.2067 | 0.3790                                        | 0.001                                         | 0.9677 |
|                   | #04                                   | 0.7899                        | 0.5588 | 2.2666 | 0.3711                                        | 0.001                                         | 0.9585 |
|                   | #05                                   | 0.3931                        | 0.7116 | 3.4675 | 0.3750                                        | 0.001                                         | 0.9930 |
|                   | Fitting considering all data together | 0.7047                        | 0.5844 | 2.4064 | 0.3793                                        | 0.001                                         | 0.9377 |
| Diesel experiment | #06                                   | 1.1497                        | 0.7439 | 3.9052 | 0.3730                                        | 0.0171                                        | 0.9758 |

## 4. Conclusions

The infiltration experiments performed demonstrated the applicability of the instrumented channel to study multiphase flow in unsaturated soils. Aspects such as the use of tensiometers to measure the suction of water and diesel at the wetting front, the visual monitoring of the infiltration experiments and the great reproducibility of the developed experimental protocol are highlights of this research.

The experimental results for water and diesel were consistent with the theory of flow in non reactive granular soils. The liquid contribution for the flow is basically given by the fluid mobility as established by Nutting (1934). The average ratio between the time required for the diesel wetting front to reach a tensiometer, to that for water, was about 8.8. This result is slightly higher than the liquid mobility ratio  $[(\rho_w/\rho_w)/(\rho_{diesel}/\rho_{diesel}) = 5.5]$ . The most probable explanation for this discrepancy is the fact that the spreading of the wetting front in the case of the diesel experiment is higher than that of water, requiring a higher infiltration volume in the soil surface to achieve the same depth below the surface.

A good adherence was observed between the experimental and predicted results ( $R^2 = 0.967$  for water and  $R^2 = 0.956$  for diesel) when the infiltration process was modeled with the uni-dimensional infiltration equation proposed by Philip (1969). The use of the saturated value of permeability led to an overestimation of the wetting front depth both for water and diesel. This is probably due to the fact that in the case of the Philip's equation a piston flow scenario is considered, which is contrary to what was observed experimentally in this paper. In order to take into account the occurrence of the drainage process in the performed tests a new value of  $k$  was adopted considering the average value of soil suction during the drainage process. In the case of the water experiments, the value of  $k_{wunsat}$  was able to reproduce the experimental data quite well. In the case of the diesel experiment, however, the value of  $k_{dunsat}$  still overestimated the experimental results. Again, the most probable explanation for this discrepancy is the higher lateral spreading of the wetting front in the case of the diesel experiment.

## Notation

$h_f$  = suction head at the wetting front, [L]  
 $h_p$  = hydrostatic head at the soil surface, [L]  
 $I(t)$  = water infiltration, [L]  
 $k$  = coefficient of permeability, [LT<sup>-1</sup>]  
 $k_w$  = water saturated coefficient of permeability, [LT<sup>-1</sup>]  
 $k_{wfit}$  = best fit for water coefficient of permeability, [LT<sup>-1</sup>]  
 $k_{wunsat}$  = unsaturated water coefficient of permeability, [LT<sup>-1</sup>]  
 $k_{diesel}$  = diesel coefficient of permeability, [LT<sup>-1</sup>]  
 $k_{dfit}$  = best fit for diesel coefficient of permeability, [LT<sup>-1</sup>]  
 $k_{dunsat}$  = unsaturated diesel coefficient of permeability, [LT<sup>-1</sup>]

$L(t)$  = wetting front position, [L]  
 $m$  = fitting parameter van Genuchten model (1980), [–]  
 $n$  = fitting parameter van Genuchten model (1980), [–]  
 $PV_{diesel}$  = diesel vapor pressure, [ML<sup>-1</sup>T<sup>-2</sup>]  
 $PV_w$  = water vapor pressure, [ML<sup>-1</sup>T<sup>-2</sup>]  
 $S_{diesel/w}$  = diesel solubility in water  
 $S$  = sorptivity, [LT<sup>-1/2</sup>]  
 $TVS$  = total volatile solids, [MM<sup>-1</sup>]  
 $\alpha$  = fitting parameter van Genuchten model (1980); [LT<sup>2</sup>M<sup>-1</sup>]  
 $\epsilon_{diesel}$  = diesel dielectric constant [–]  
 $\epsilon_w$  = water dielectric constant [–]  
 $\mu_{diesel}$  = diesel liquid viscosity, [ML<sup>-1</sup>T<sup>-1</sup>]  
 $\mu_w$  = water liquid viscosity, [ML<sup>-1</sup>T<sup>-1</sup>]  
 $\psi$  = soil suction, [L]  
 $\psi_b$  = bubbling pressure, [L]  
 $\rho_b$  = soil bulk density, [ML<sup>-3</sup>]  
 $\rho_s$  = grain density, [ML<sup>-3</sup>]  
 $\rho_{diesel}$  = diesel liquid density, [ML<sup>-3</sup>]  
 $\rho_w$  = water liquid density, [ML<sup>-3</sup>]  
 $\sigma_{diesel}$  = diesel surface tension, [MT<sup>-2</sup>]  
 $\sigma_w$  = water surface tension, [MT<sup>-2</sup>]  
 $\theta$  = liquid volumetric content, [L<sup>3</sup>L<sup>-3</sup>]  
 $\theta_s$  = liquid volumetric content at complete saturation, [L<sup>3</sup>L<sup>-3</sup>]  
 $\theta_r$  = liquid volumetric content at residual saturation, [L<sup>3</sup>L<sup>-3</sup>]  
 $\Theta$  = effective liquid volumetric content, [–]  
 BTEX = benzene, toluene, ethyl-benzene and xylene  
 DNAPL = dense non-aqueous phase liquid  
 EMBASA = Empresa Baiana de Água e Saneamento  
 GPR = ground penetrating radar  
 LNAPL = light non-aqueous phase liquid  
 NAPL = non-aqueous phase liquid  
 NBR = Brazilian technical standard  
 SUCS = unified system of soil classification  
 SLRC = soil liquid retention curve  
 SWRC = soil water retention curve  
 VO = visual observations  
 TR = tensiometer reading  
 2D = two-dimensional  
 3D = three-dimensional

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# Evaluation of the Aggregate Produced From Wastes of Quartzite Mining Sites to Use in Concrete

E.G. Collares, I. Francklin Jr., L.A.C. Motta

**Abstract.** The quartzite found in rock formations in the Southwestern Minas Gerais - Brazil has been, along the years, explored and commercialized as “coating stone”. The quantity of waste coming from the processing and exploration process, however, is superior to 90% from the extracted product in most of the mining sites, determining a serious environmental passive. This work presents results which can motivate the alternative use of the materials that are dumped on piles of waste. The research involved a petrographic analysis of quartzite occurring in the principal mines of the region; evaluation of aggregates produced in two kinds of crushers; evaluation of the physical and mechanical properties of the rock and evaluation of the physical and mechanical properties of the concrete manufactured with the quartzite aggregate. The results of the technological tests carried out on the rock material were satisfactory and meet all the specifications for the use in concrete. The samples of concrete prepared with the quartzite aggregate presented results of strength similar to the ones manufactured with the aggregate which are used commercially in the region today.

**Keywords:** quartzite, mining waste, aggregate for construction, concrete.

## 1. Introduction

The Southwestern Minas Gerais is known nationally by the production of quartzites used as the civil construction coating, which is called “pedra mineira”. The extraction of the quartzite in the region occur, many times, on a clandestine basis and/or not meeting the requirements necessary for the environmental control of that activity.

Even the mining sites which exert their activities legally correct, obeying the specifications exposed in the environmental report approved by environmental bureaus, the quantity of waste generated in the extraction process, the so-called “mine dump” sites, is very big. According to information given by environmental bureaus, this waste may get to 92% of the extracted material. This happens because the quartzite is used, basically, as coating stone and thus, should be extracted in plaques, obeying width and length standards. All the material extracted not obeying this standard is disposed as “waste”.

All the waste generated in the processing and extract process of the quartzite turns into a big problem to businessmen, because, due to the great quantity of material, it begins producing negative environmental impacts, such as: landscape disfiguration, alteration in the natural formation of reliefs, collapsing the water bodies, supplying the native vegetation, stabilizations on the slopes, to mention a few.

On the other hand, in case that waste obeys the normative standard established for the use of rock material in different possibilities of use in the civil construction, it can

be seen as material feasible for other purposes, offering, thus, alternatives to exclusive use as a coating stone.

One of the alternatives could be the use of the waste of the quartzite as aggregate in the civil construction, as component material of concrete, railway or road buildings. Collares *et al.* (2008) present a paper in which they study rock material used as aggregate in the Southwestern Minas Gerais and they include a quartzite sample in their analysis, and in a previous evaluation, they describe that this lithological kind presents results similar to those which are used commercially in the region (diabase, gneiss and limestone).

This present paper presents results of a technological study carried out in material dumped as waste in mine dumps in quartzite mines of five municipalities of the Southwestern Minas Gerais and in concrete test specimen manufactured with aggregate obtained from these materials.

Few studies which involve the use of the quartzite as an aggregate in the civil construction are available in Brazil. Andrade (1997) studied several types of quartzite from different origins. The results presented correspond to the compressive strength, specific density and water absorption. Strength values varying from 54 MPa to 367 MPa and absorption almost always inferior to 1% were found.

Sbrighi Neto (2005) presents typical values of some properties of gneisses and quartzites used in the production of aggregate to concrete (Table 1). It is observed a high

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**Table 1** - Some typical properties of gneisses and quartzite for the use as aggregate in concrete (Sbrighi Neto, 2005).

| Rock      | Compressive strength (MPa) |         |         | Absorption (%) | Specific density (g/cm <sup>3</sup> ) | Deformation modulus (GPa) |
|-----------|----------------------------|---------|---------|----------------|---------------------------------------|---------------------------|
|           | Average                    | Maximum | Minimum |                |                                       |                           |
| Gneiss    | 150                        | 240     | 100     | 0.2 a 0.8      | 2.55 a 2.70                           | 40 a 70                   |
| Quartzite | 260                        | 400     | 130     | 0.1 a 0.8      | 2.55 a 2.70                           | 50 a 100                  |

variability for the compressive strength and low variability for absorption.

A very important aspect to be evaluated when using the quartzite in concrete is the reactivity. The Alkali-Aggregate Reaction (AAR) according to Hasparyk (2005) can be defined as a chemical reaction which occurs in a concrete structure, involving the alkaline hydroxide coming principally, from cement and some reactive minerals present in the aggregate used. As a result from this reaction, products are formed, which in the presence of humidity, are able to expand, generating fissures, deviation, and may endanger the concrete structure.

Hasparyk (2005) analyzed the concrete of a structure from the dam at the Hydroelectric Power Plant in Furnas in São José da Barra, in Southwestern Minas Gerais, produced with quartzite aggregate. That dam, constructed between 1950 and 1963, has already presented problems coming from AAR. The study consisted of an investigation with witnesses of concrete checking the properties in the hardened state, micro-structural analysis of the concrete and of the quartzite aggregate to investigate the deleterious reactions.

There are different laboratory methods to determine the deleterious potentiality of the aggregate combined with cement alkalis and according to Hasparyk (1999) the most common are: petrographic analysis, Osipov Test (thermal), Chemical Test, bar test, accelerated test, carbon rock test, concrete prism test and scanning electronic microscopy. One of the most common tests carried out in laboratories to check the deleterious potentiality of aggregates is performed by the accelerated test, which uses bars of mortar in alkaline solution. This method is used in this study.

## 2. The Quartzites in the Southwestern Minas Gerais

According to COMIG (1994), the geological formations that occur in the Southwestern Minas Gerais are the following:

- Araxá Group: schists, amphibolites, quartzites, gneisses and iron formation;
- São Bento Group: (Serra Geral Formation): basalts and diabbases;
- Canastra Group: quartzites, phyllites and schists;
- Carmo do Rio Claro Group: carbonaceous or not carbonaceous phyllites and quartzites;

- Campos Gerais Complex: banded gneisses, migmatites, granite-gneisses, amphibolites, quartzites, schists, meta-ultramafics.

The quartzites of the groups Araxá and Canastra were used in this study, which arise in the Mid-North of the Southwestern Minas Gerais and are used as “coating stones”. A survey of the principal mines that are exploring this quartzite was conducted. The studies went forward to mining sites located in the municipalities of São João Batista do Glória, Capitólio, São José da Barra, Alpinópolis and Guapé.

### 2.1. Preliminary study to select the study places

This preliminary study was done with the purpose of selecting samples to carry out the most specific technological studies, which constitute the subject matter of the research. Table 2 presents the information concerning the samples collected in the mines visited.

Soon after a petrographic approval (macroscopic analysis) in each lithological kind, according to ABNT (1992a), it was observed that the samples selected in each mine present some particularities concerning the texture, structure, and mineralogical composition. It was observed in almost all the mines, two types of quartzite: Type 1: white-yellowish and varied, foliated, with medium mica content (muscovite) and it is used as “coating stone”; Type 2: gray, pretty silicified, with lower muscovite content and less evident foliation. Type 2 is not used as “coating stone” and constitute of residues in mines.

In order to verify the mineralogical and structural differences of Quartzite types 1 and 2, a microscopic analysis (in thin sections) of the selected samples was performed.

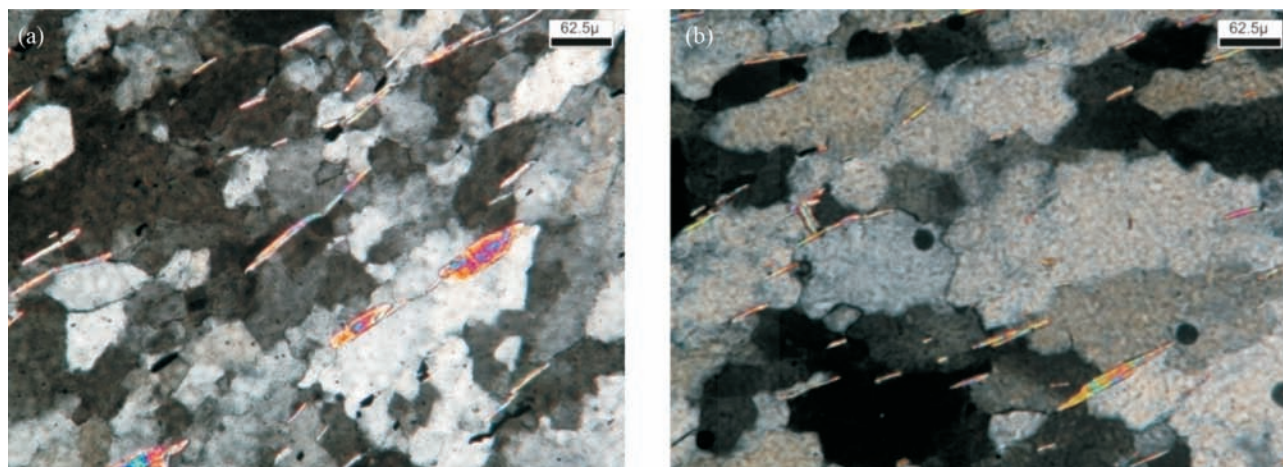
The thin sections were made in the Rock Laboratory at the Technological Center of Civil Engineering, located in Aparecida de Goiânia - GO at Furnas Centrais Elétricas S/A. The descriptions of the thin sections were performed by the Rock laboratory at UNESP - Rio Claro Campus - SP and confirmed the existence of two types of quartzite (Types 1 and 2). Type 1 presents a little higher mica content (around 4%) and more emphasized foliation (Fig. 1 and Table 3). Further details about the quartzite petrographic characteristics can be checked in Francklin Jr. (2009).

### 2.2. Selection of higher strength samples

All samples collected were received tests with the objective of measuring the strength to rock rupture using the Method for Determining Point Load Strength -  $I_{s(50)}$  (ISRM,

**Table 2** - Location of the mines visited.

| Mining | Altitude (m) | Latitude     | Longitude    | Sample              |
|--------|--------------|--------------|--------------|---------------------|
| ALP 1  | 1100         | 20°51'05'' S | 46°21'14'' W | 5-A-A e 5-A-B       |
| ALP 2  | 1026         | 20°51'17'' S | 46°21'36'' W | 2-A-A e 2-A-B       |
|        | 1049         |              |              | 3-A-A e 3-A-B       |
|        | 1106         |              |              | 1-A                 |
|        | 1110         |              |              | 4-A                 |
| SJB 1  | 919          | 20°42'33'' S | 46°17'48'' W | 7-A                 |
| SJB 2  | 1084         | 20°42'14'' S | 46°17'17'' W | 6-A-A e 6-A-B       |
| GLO 1  | 1021         | 20°37'12'' S | 46°17'30'' W | 1-G                 |
| GLO 2  | 1126         | 20°36'17'' S | 46°18'42'' W | 3-G-A e 3-G-B       |
| CAP 1  | 1044         | 20°37'53'' S | 46°16'44'' W | 5-G                 |
| CAP 2  | 1216         | 20°34'57'' S | 46°16'50'' W | 4-G-A e 4-G-B       |
| CAP 3  | 1324         | 20°34'47'' S | 46°17'34'' W | 2-G-A 2-G-B e 2-G-C |
| CAP 4  | 1285         | 20°35'11'' S | 46°17'59'' W | 7-G-A e 7-G-B       |
| CAP 5  | 1233         | 20°36'36'' S | 46°16'26'' W | 6-G-A e 6-G-B       |
| GUA    | 898          | 20°50'03'' S | 45°55'43'' W | A5                  |

**Figure 1** - (a) Microscopy quartzite Type 1; (b) Microscopy quartzite Type 2.**Table 3** - Petrographic description of the quartzites types 1 and 2 (analyses of the Geociências Department of the UNESP, Rio Claro, SP).

| Samples          | Modal composition*                                                                                                                                       | Classification                         |
|------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------|
| Quartzite Type 1 | Quartz 95%<br>Muscovite 4%<br>Opaque Minerals %<br>Turmaline - occasional occurrence<br>Zircon - occasional occurrence<br>Rutile - occasional occurrence | Muscovite Quartzite schistose          |
| Quartzite Type 2 | Quartz 97%<br>Muscovite 1%<br>Opaque Minerals 1%<br>Turmaline %<br>Zircon - occasional occurrence<br>Rutile - occasional occurrence                      | Foliated Quartzite with Muscovite thin |

\* visually estimated.

1985). The objective was to select the most resistant samples.

In Fig. 2 it is possible to check the difference of the rupture loads, between Types 1 and 2 rocks. It is clear the higher strength to the samples Type 2 (gray) when they are submitted to the application of compression strength applied directly to the samples.

The samples which presented high values of rupture, described as Type 2 in the petrographic analysis, are exactly those rejected in the extraction of mines and were the specific subjects in this paper.

Samples for the specific studies (technological tests in aggregate and tests in the concrete produced with the aggregate) were chosen based on the preliminary study and were collected at dumps of mines in the coordinates presented in Tables 4 and 5.

In Phase 1 the samples were named A1, A2, A3 and A4, corresponding to the cities of São João Batista do

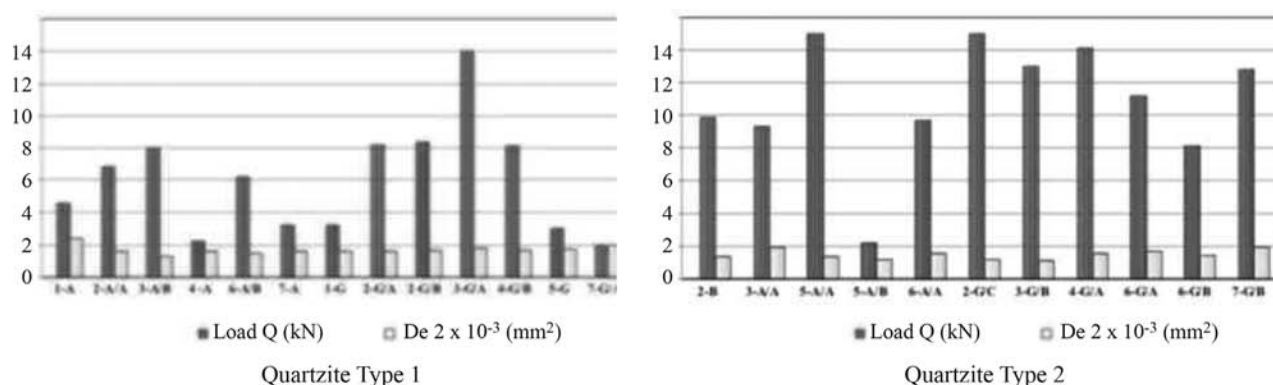
Glória, Capitólio, São José da Barra and Alpinópolis respectively. In Phase 2 only in São João Batista do Glória the collection of samples was not done due to technical problems and it was replaced by mines in the municipality of Guapé. The samples collected in the second phase were named A2 - Phase 2 (Capitólio), A3 - Phase 2 (São José da Barra), A4 - Phase 2 (Alpinópolis) and A5 - Phase 2 (Guapé). The sample of gneiss was used as a reference because it is used commercially in the region as an aggregate. This sample was called AG and was collected in the municipality of Passos - MG.

### 3. Methodology

#### 3.1. Sample collection and aggregate production

The collections were carried out in two phases:

Phase 1 (small sample volume): the samples were selected and collected manually at mine dumps. Samples of



**Figure 2** - Graph showing the rupture and sample areas of Type 1(a) and 2 (b). “De” refers to the diameter of the sample.

**Table 4** - Coordinates of the samples in Phase 1.

| Phase 1                    |              |              |              |                 |
|----------------------------|--------------|--------------|--------------|-----------------|
| Municipalities             | Altitude (m) | Latitude     | Longitude    | Sample          |
| São João Batista do Glória | 1126         | 20°36'17'' S | 46°18'42'' W | <b>A1</b> - GLO |
| Capitólio                  | 1324         | 20°34'47'' S | 46°17'34'' W | <b>A2</b> - CAP |
| São José da Barra          | 1084         | 20°42'14'' S | 46°17'17'' W | <b>A3</b> - SJB |
| Alpinópolis                | 1026         | 20°51'17'' S | 46°21'36'' W | <b>A4</b> - ALP |

**Table 5** - Coordinates of samples in Phase 2.

| Phase 2           |              |                 |                 |                 |
|-------------------|--------------|-----------------|-----------------|-----------------|
| Municipality      | Altitude (m) | Latitude        | Longitude       | Sample          |
| Capitólio         | 1188         | 20°37'3.13'' S  | 46°15'3.68'' W  | <b>A2</b> - CAP |
| São José da Barra | 1072         | 20°42'6.25'' S  | 46°17'10.26'' W | <b>A3</b> - SJB |
| Alpinópolis       | 1019         | 20°50'53.11'' S | 46°21'15.88'' W | <b>A4</b> - ALP |
| Guapé             | 947          | 20°50'3.19'' S  | 45°55'43.72'' W | <b>A5</b> - GUA |

Type 2 quartzite were selected. The samples were collected in the municipalities of São João Batista do Glória, Capitólio, São José da Barra and Alpinópolis and so-called: A1, A2, A3 and A4.

Phase 2 (big sample volume): The mines where the sample collection was performed were the same of the previous phase (except São João Batista do Glória, which was replaced by Guapé - sample A5). During this phase a big volume of sample was collected (around 4 tons), direct from mine dumps, with the help of a wheel loader and a dump truck was used for transportation.

With a good quantity of collect it was possible the crushing in an outstanding crushing site, which permitted to obtain more regular samples (those which fit better with the commercial product) and an analysis of the crushing process.

The samples were crushed in two different crushers: the jaw crusher and hydrocone crusher. The crusher specifications are in Table 6.

### 3.2. Preliminary tests with the crushing material

The preliminary tests performed were: Grain-size Analysis (ABNT, 2003b), whose final results were compared to the aggregate quality parameters established by ABNT (2005); Shape Index (ABNT, 1983a and ABNT, 1989) and Unit Mass in Loose State (ABNT, 1982) and Compact State (ABNT, 1983b).

### 3.3. Technological tests in aggregate

In order to achieve a technological characterization of the quartzite aggregates the following tests were performed: Dry Density; Density in Saturated and Surface-dry Conditions; Absorption and Porosity (ABNT, 2002); Artificial Water/Stove Cycling (ABNT, 1992b); Powder Material Tenor (ABNT, 2001), "Los Angeles" Abrasion (ABNT, 2000); Crushing (ABNT, 1987); Uniaxial Compression (ABNT, 2007) and Potential Alkali Reactivity of Aggregates (ASTM, 2007).

### 3.4. Concrete manufacturing

With the results of the tests for material characterization in hands, concrete was manufactured according to ABNT (1993), analyzing the behavior in fresh and hardened state.

The concrete production was divided into two phases, the first one using processed aggregates in a Jaw Crusher (Phase 1) and the second one using the processed aggregates in a Hydrocone Crusher (Phase 2).

The main purpose was to analyze the quartzite aggregate behavior in concrete, both in fresh and hardened state.

For the production of the reference concrete traditional material was used such as CP II-Z-32 cement, river sand and gneiss crushing (the latter was used as a comparative reference). For the production of quartzite concrete of each sample, the gneiss used as coarse aggregate was replaced by the quartzite samples.

In order to have a coherent comparative analysis between the results of the concrete properties, the same processes of crushing and grain size classification to constitute the gneiss coarse aggregates and quartzite were used.

The reference trace was chosen with the intention of comparing its results with the other concretes produced with coarse aggregate of five different types of quartzite. The dosage study of this trace was carried out through the ACI/ABCP method, which aims at obtaining ideal minimum consume of mortar content, coming from the principle of the specific area analysis of the fine aggregate and from the smallest empties index of the coarse aggregate, with the objective of reducing the water/cement (w/c) ratio.

The representation of the reference trace chosen in dry material mass was 1 : 1.95 : 2.9 : 0.57 in Phase 1 e 1 : 1.95 : 2.9 : 0.55 in Phase 2.

For all the traces the w/c ratio was kept with the purpose of checking possible interferences of quartzite aggregate in the workability and consistency of concretes. Tables 7 and 8 show the abbreviations, the rock types, and the origin of the coarse aggregates, referring to the traces studied in Phases 1 and 2, respectively.

Seven 15 cm x 30 cm cylindrical specimens were casted for each of the five traces studied, aiming at testing at least two samples of the same concrete with 28 days for the mechanical tests proposed.

**Table 7** - Identification of the concretes studied with reference to the coarse aggregate in Phase 1.

| Trace denomination | Coarse aggregate  | Origin of the coarse aggregate |
|--------------------|-------------------|--------------------------------|
| AG                 | Gneiss            | Passos/MG                      |
| A1                 | Quartzite - GLO 2 | S. J. B. Glória/MG             |
| A2                 | Quartzite - CAP 3 | Capitólio/MG                   |
| A3                 | Quartzite - SJB 2 | S. J. Barra/MG                 |
| A4                 | Quartzite - ALP 2 | Alpinópolis/MG                 |

**Table 6** - Crusher specifications used in this research.

| Crusher | Type      | Model  | Manufacturer | Size (cm)                | Processing (T/h) |
|---------|-----------|--------|--------------|--------------------------|------------------|
| Phase 1 | Jaw       | 02     | Marumby      | 200(c) x 140(l) x 110(e) | 10               |
| Phase 2 | Hydrocone | H 2000 | Sandvik      | 216(h) x 980(Ø)          | 80               |

**Table 8** - Identification of the concretes studied with reference to the coarse aggregate in Phase 2.

| Trace denomination | Coarse aggregate | Origin of the coarse aggregate |
|--------------------|------------------|--------------------------------|
| AG (Phase 2)       | Gneiss           | Passos/MG                      |
| A2 (Phase 2)       | Quartzite - CAP  | Capitólio/MG                   |
| A3 (Phase 2)       | Quartzite- S.J.B | S. J. Barra/MG                 |
| A4 (Phase 2)       | Quartzite- ALP   | Alpinópolis/MG                 |
| A5 (Phase 2)       | Quartzite - GUA  | Guapé/MG                       |

The concrete properties in the hardened state analyzed in this paper were:

- Compressive strength (ABNT, 2007);
- Tensile Strength by diametral compression (ABNT, 1994).
- Modulus of elasticity (ABNT, 2003a), performed in Phase 2.

## 4. Results and Discussions

### 4.1. Technological characterization of gneiss and quartzite aggregates

The results are compared with the limits established by ASTM and ABNT, the guide for assessing the quality of the aggregate (Table 9) based on results of laboratory tests performed by Verhoef and Van De Wall (1998, apud Gomes, 2001) and studies of quartzite and other wastes for use in construction.

#### 4.1.1. Shape index

Tables 10 and 11 show the results of the Shape Index Tests in Phases 1 and 2, respectively.

The results of the shape index presented in Phase 1 have confirmed the low uniformity of the jaw crusher, when producing irregular grains. Only samples A2 and AG have formed regular grains according to ABNT (1983a). The use of Hydrocone Crusher in Phase 2 has shown effective in producing cubic grains of aggregate and c/e ratio lower than 3, essential to produce high compactness concrete.

#### 4.1.2. Density, water absorption and porosity rate

The results of Density, Water Absorption and Porosity Rate are presented in Table 12 for the aggregate processed in Phase 1 and in Table 13 for the aggregate processed in Phase 2.

The results of density (dry and Saturated and Surface-dry Conditions), for all the samples were satisfactory, with very small variations among the values.

Values between 0.5% e 2% are considered good absorption and values lower than 0.5% are considered excellent absorption according to the parameters established by Verhoef and Van De Wall (1998 *apud* Gomes, 2001), therefore in Phase 1 only sample A3 in the municipality of São José da Barra was considered good and all the others were considered excellent. In Phase 2 all the samples were considered to be excellent.

According to the parameters established by Verhoef and Van De Wall (1998 *apud* Gomes, 2001), values be-

**Table 9** - Guidance for assessing the quality of the aggregate based on results of laboratory (Verhoef & Van De Wall, 1998 apud Gomes, 2001).

|                            | Excellent | Good    | Tolerable | Poor  |
|----------------------------|-----------|---------|-----------|-------|
| Water absorption (%)       | > 0.5     | 0.5-2.0 | 2.0-6.0   | < 6.0 |
| Porosity rate (%)          | > 2       | 2-3     | 3-4       | < 4   |
| Crushing (%)               | > 20      | 20-25   | 25-30     | < 30  |
| “Los Angeles” abrasion (%) | > 40      | 40-45   | 45-50     | < 50  |

**Table 10** - Aggregate shape index in Phase 1.

| Samples | Phase 1            |      |       |               |            |
|---------|--------------------|------|-------|---------------|------------|
|         | ABNT NBR 6954:1989 |      |       | NBR 7809:1993 |            |
|         | B/A                | C/B  | Form  | C/E           | Condition  |
| AG      | 0.67               | 0.59 | Cubic | 2.66          | Acceptable |
| A1      | 0.58               | 0.65 | Cubic | 3.14          | Rejected   |
| A2      | 0.57               | 0.59 | Cubic | 2.8           | Acceptable |
| A3      | 0.6                | 0.58 | Cubic | 3.7           | Rejected   |
| A4      | 0.54               | 0.63 | Cubic | 3.57          | Rejected   |

**Table 11** - Aggregate index form in Phase 2.

| Samples | Phase 2            |      |       |               |            |
|---------|--------------------|------|-------|---------------|------------|
|         | ABNT NBR 6954:1989 |      |       | NBR 7809:1993 |            |
|         | B/A                | C/B  | Form  | C/E           | Condition  |
| AG      | 0.74               | 0.69 | Cubic | 2             | Acceptable |
| A2      | 0.7                | 0.63 | Cubic | 2.68          | Acceptable |
| A3      | 0.64               | 0.62 | Cubic | 2.52          | Acceptable |
| A4      | 0.67               | 0.59 | Cubic | 2.79          | Acceptable |
| A5      | 0.66               | 0.64 | Cubic | 2.81          | Acceptable |

**Table 12** - Density, water absorption and porosity rate of aggregates in Phase 1.

| Samples | Phase 1                          |                                                                      |                      |                   |
|---------|----------------------------------|----------------------------------------------------------------------|----------------------|-------------------|
|         | Dry density (kN/m <sup>3</sup> ) | Density in saturated and surface-dry conditions (kN/m <sup>3</sup> ) | Water absorption (%) | Porosity rate (%) |
| AG      | 27.4                             | 27.5                                                                 | 0.3                  | 0.8               |
| A1      | 26.3                             | 26.4                                                                 | 0.4                  | 1.02              |
| A2      | 26.4                             | 26.5                                                                 | 0.2                  | 0.57              |
| A3      | 25.9                             | 26.1                                                                 | 0.9                  | 2.30              |
| A4      | 26.2                             | 26.3                                                                 | 0.5                  | 1.32              |

**Table 13** - Density, water absorption and porosity rate of aggregates in Phase 2.

| Samples | Phase 2                          |                                                                      |                      |                   |
|---------|----------------------------------|----------------------------------------------------------------------|----------------------|-------------------|
|         | Dry density (kN/m <sup>3</sup> ) | Density in saturated and surface-dry conditions (kN/m <sup>3</sup> ) | Water absorption (%) | Porosity rate (%) |
| AG      | 27.2                             | 27.3                                                                 | 0.3                  | 0.8               |
| A2      | 26.2                             | 26.4                                                                 | 0.5                  | 1.4               |
| A3      | 26.2                             | 26.4                                                                 | 0.5                  | 1.4               |
| A4      | 26.5                             | 26.7                                                                 | 0.5                  | 1.4               |
| A5      | 26.3                             | 26.4                                                                 | 0.4                  | 1.1               |

tween 2.0 and 3% are considered good porosity rate and values that are lower than 2% are considered excellent. In Phase 1 only sample A3 was classified as a good porosity rate and the other samples were classified as excellent. In Phase 2 all the samples were classified as excellent, with values lower than 2%.

#### 4.1.3. Artificial water/stove cycling

Table 14 shows the percentages of mass loss regarding the initial mass of the samples determined during the quantitative exam.

During each cycle it was observed whether the fragments underwent any kind of attack due to the presence of expansive clay mineral, such as disintegration, rupture, crack and spalling. The sample that presented

**Table 14** - Verification of aggregate mass loss after the artificial water/stove cycling test.

| Sample | Initial mass (g) | Final mass (g) after 120 cycles | Loss mass in % |
|--------|------------------|---------------------------------|----------------|
| AG     | 470.2            | 468.4                           | 0.38%          |
| A1     | 317.3            | 315.6                           | 0.54%          |
| A2     | 350.1            | 348.7                           | 0.40%          |
| A3     | 526.0            | 524.2                           | 0.34%          |
| A4     | 497.3            | 496.1                           | 0.24%          |
| A5     | 573.3            | 571.4                           | 0.33%          |

more mass loss was that of A1, but it was not meaningful. As it was expected none of them showed presence of ex-

pansive minerals, not suffering, thus, degradations mentioned before.

#### 4.1.4. Grain size analysis

When the jaw crusher was used in Phase 1, only the fragmentation of the material was performed. The samples were fragmented in varied dimensions and taken to the laboratory for classification according to ABNT (2005), using normal and intermediate sieves. All aggregate samples received the same classification process in order to have the same granulometric profile. The grain size analysis results and the limits of the granulometric zones  $d/D = 9.5/25$  of A1 and AG samples processed in jaw crusher (Phase 1) are shown in Fig. 3. Samples had the same classification profile with a continuous granulometric distribution and next to the interval of limits established by ABNT (2005).

The samples processed by the hydrocone crusher were classified by the equipment itself after the secondary crushing. For the concrete production fractions of 19 mm were chosen, commercialized as crushed stone 1.

The results of the tests performed in laboratories and the limits of the granulometric zone  $d/D = 9.5/25$  according

to ABNT (2005) of each sample processed in hydrocone crusher are shown in Fig. 4.

The results of the samples processed and classified in the hydrocone crusher (Phase 2) fit the interval of limits (inferior and superior) established by ABNT (2005) to be used as coarse aggregate in the concrete.

#### 4.1.5. Unit mass

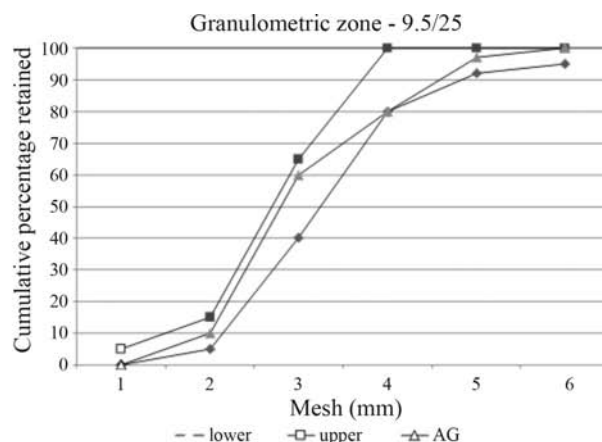
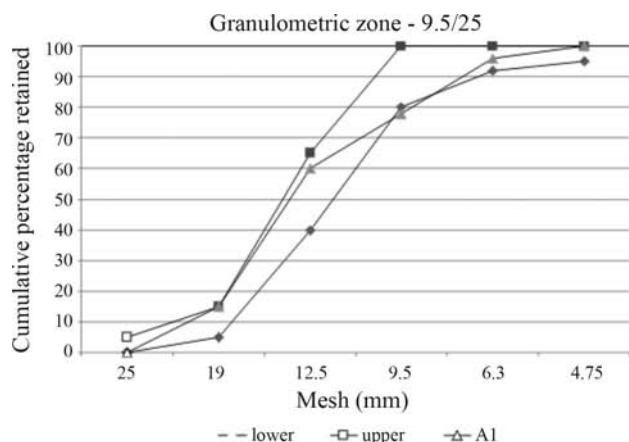
In order to have the results of unit mass in loose and compacted states the samples processed in both crushers were used (Phases 1 and 2).

##### a) Unit mass in loose state

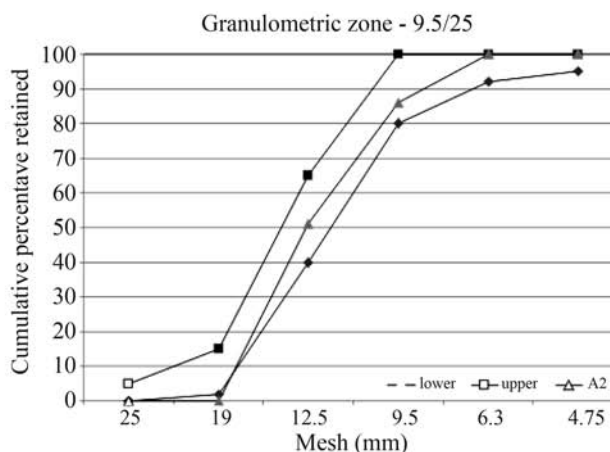
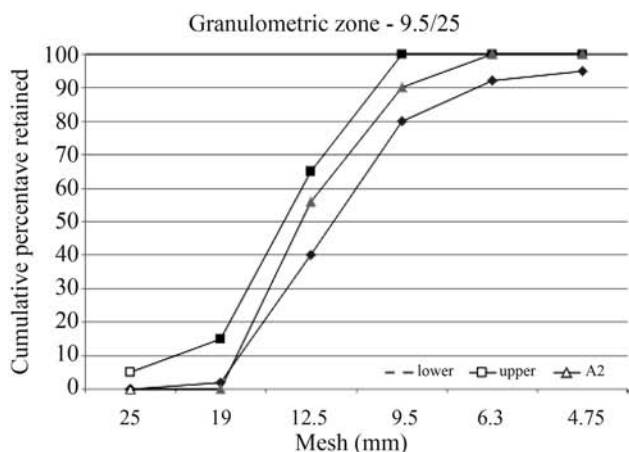
Tables 15 and 16 show the results of the unit mass of quartzite and gneiss aggregates in loose state with their respective void ratio in Phases 1 and 2. The results obtained of unit mass of samples processed in Phase 2 were better regarding the samples processed in Phase 1, proving once again the efficiency of the hydrocone crusher which contributed to the reduction of the void ratio of the aggregates.

##### b) Unit mass in compacted state

Tables 17 and 18 show the results of the unit mass of quartzite aggregates in compacted state in Phases 1 and 2,



**Figure 3** - Grain size analysis of quartzite samples A1 (a) and Gneisses - AG (b) processed in Phase 1, in granulometric zone 9.5/25.



**Figure 4** - Grain size analysis of quartzite samples A2 (a) and AG (b) processed in Phase 2, in granulometric zone 9.5/25.

**Table 15** - Results of the unit mass of aggregates in loose state in Phase 1.

| Phase 1 |                                               |            |
|---------|-----------------------------------------------|------------|
| Sample  | Unit mass in loose state (kN/m <sup>3</sup> ) | Void ratio |
| AG      | 14.80                                         | 46.00%     |
| A1      | 13.63                                         | 48.20%     |
| A2      | 13.69                                         | 48.10%     |
| A3      | 13.61                                         | 48.10%     |
| A4      | 13.63                                         | 48.00%     |

**Table 16** - Results of the unit mass of aggregates in loose state in Phase 2.

| Phase 2 |                                               |            |
|---------|-----------------------------------------------|------------|
| Sample  | Unit mass in loose state (kN/m <sup>3</sup> ) | Void ratio |
| AG      | 14.53                                         | 46.60%     |
| A2      | 14.82                                         | 43.90%     |
| A3      | 14.47                                         | 44.80%     |
| A4      | 14.25                                         | 45.60%     |
| A5      | 14.55                                         | 44.70%     |

respectively. Values very close to the unit mass in compacted state were found among the samples processed in the different crushers. As expected, the samples processed in Phase 1 with the jaw crusher presented inferior values to the ones processed in Phase 2 with the hydrocone crusher.

#### 4.1.6. Powdery material content

ABNT (2005) determines that the maximum quantity of powdery materials that the coarse aggregate to use in concrete may have is 1%. According to Table 19, of all the five crushed samples, only the AG sample presents unfavorable condition. But ABNT NBR 7211:2005 allows a tolerance to the rocks with water absorption rate inferior to 1%, determined according to ABNT (2001). Thus, the limit of fine material (powdery) becomes 2%. This applies to AG sample, which has an absorption rate of 0.3%.

#### 4.1.7. “ Los Angeles ” abrasion

ABNT (2005) establishes that the abrasive wearing off underwent by the aggregate according to ABNT (2000) must be inferior to 50% in mass. Figure 5 shows that samples in Phases 1 and 2 presented satisfactory behavior of the aggregate wearing off to abrasion.

#### 4.1.8. Crushing

It is possible to verify in Fig. 6(a) that the results of the crushing strength of samples A2 and A4, in Phase 1, were classified as excellent, with loss percentages lower than 20%. The samples of quartzite A1 and A3 fit the inter-

**Table 17** - Unit mass of quartzite aggregates in compacted state - Phase 1.

| Phase 1 |                                                   |
|---------|---------------------------------------------------|
| Sample  | Unit mass in compacted state (kN/m <sup>3</sup> ) |
| A2      | 15.17                                             |
| A3      | 15.11                                             |
| A4      | 15.03                                             |
| A5      | 15.20                                             |

**Table 18** - Unit mass of quartzite aggregates in compacted state - Phase 2.

| Phase 2 |                                                   |
|---------|---------------------------------------------------|
| Sample  | Unit mass in compacted state (kN/m <sup>3</sup> ) |
| A1      | 17.18                                             |
| A2      | 17.20                                             |
| A3      | 17.32                                             |
| A4      | 17.14                                             |

**Table 19** - Results of the powdery material content.

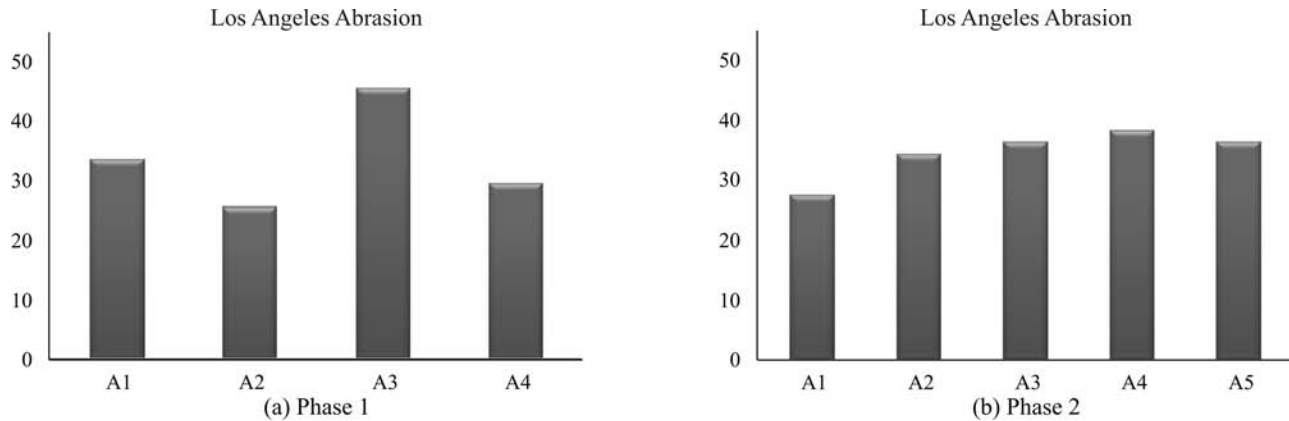
| Samples | Initial mass (g) | Final mass (g) | Powdery material (%) |
|---------|------------------|----------------|----------------------|
| AG      | 3000.3           | 2967.0         | 1.11                 |
| A1      | 3000.0           | 2982.9         | 0.57                 |
| A2      | 3000.0           | 2986.2         | 0.46                 |
| A3      | 3000.2           | 2982.1         | 0.60                 |
| A4      | 3000.3           | 2970.6         | 0.99                 |
| A5      | 3000.1           | 2983.7         | 0.55                 |

val of 20% and 25%, therefore they were classified as having a good index to use as aggregate in the civil construction, according to Verhoef e Van De Wall (1998 *apud* Gomes, 2001).

Figure 6(b) presents the results of crushing strength of aggregates in Phase 2. AG and A1 samples were classified as excellent with loss percentages lower than 20%. A3, A4, and A5 quartzite samples fit the interval of 20 and 25%, therefore they were considered good for use.

#### 4.1.9. Potential alkali reactivity of aggregates

It was produced two traces of mortar for each of the four quartzite samples (A1, A2, A3 e A4). For each trace three bars of mortar were casted, totalizing 24 bars and the expansion was measured periodically according to the accelerated test of ASTM (2007). The first trace was produced using CP II-Z-32 cement and the second trace was produced using the CP V-ARI cement. The chemical analy-



**Figure 5** - Indices of "Los Angeles" abrasion.

sis of these two kinds of cement emphasizing the alkali content is described in Table 20. The equivalent alkaline presents values practically identical in both cements.

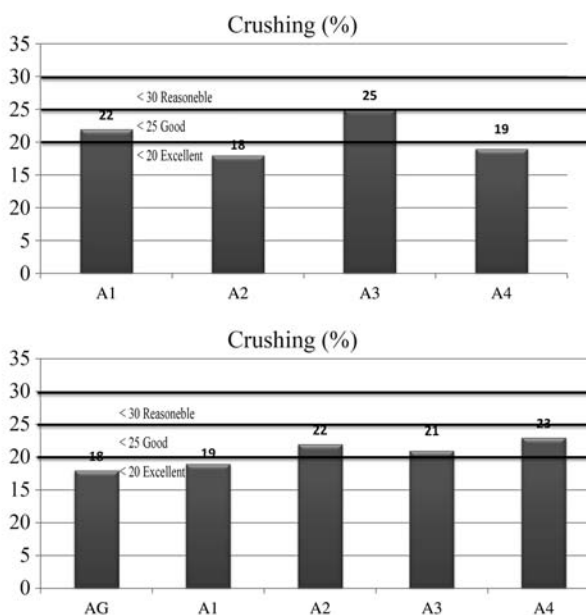
Figure 7 shows the growing behavior of mortar bar expansion up to 30 days, and Table 21 contains the values of expansion within 16 and 30 days for each kind of cement.

According to the limits of ASTM (2007) at 16 days, A1, A2, and A3 samples produced with CP II-Z-32 cement, indicated an innocuous behavior, with expansions lower than 0.10%, whereas A4 sample obtained average expansion of 0.116% indicating the possibility of deleterious manifestation. On the 30<sup>th</sup> day of testing, it was proved the potentially deleterious behavior in A4 sample, indicating expansions higher than 0.2%.

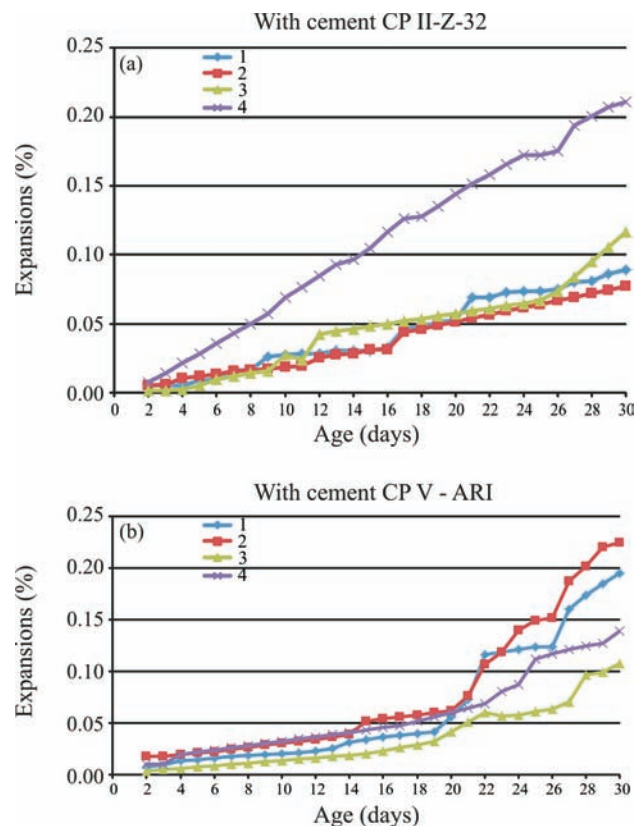
All the mortar bars produced with the CP V-ARI cement analyzed on the 16<sup>th</sup> day indicated an innocuous behavior with expansions lower than 0.10%, however, on the

30<sup>th</sup> day of testing, it was confirmed a potentially deleterious behavior in A2 sample, indicating expansions higher than 0.2%. Results indicated the possibility of the presence of aggregates with both behavior - innocuous and deleterious, in samples A1, A3, and A4. Sample A3 presented the lowest rate of expansion, but within the range of deleterious potentiality.

The results using the CP II-Z-32 presented shorter expansions in most of the mortar bars when compared to the results of the CP V-ARI. Since the alkali proportions were



**Figure 6** - Indices of crushing resistance. (a) Phase 1. (b) Phase 2.



**Figure 7** - Expansion of the mortar bars produced with the samples of quartzite aggregates.

**Table 20** - Alkaline content of CP II Z 32 and CO V-ARI cements.

| Results in % | CP II Z 32       |                   |       |      |                     | CP V ARI         |                   |       |      |                     |
|--------------|------------------|-------------------|-------|------|---------------------|------------------|-------------------|-------|------|---------------------|
|              | K <sub>2</sub> O | Na <sub>2</sub> O | CaO   | MgO  | Equivalent alkaline | K <sub>2</sub> O | Na <sub>2</sub> O | CaO   | MgO  | Equivalent alkaline |
| Sample 1     | 0.8              | 0.13              | 56.53 | 6.28 | 0.66                | 0.76             | 0.18              | 63.44 | 0.73 | 0.68                |
| Sample 2     | 0.89             | 0.12              | 60.59 | 4.3  | 0.71                | 0.74             | 0.18              | 64.22 | 0.77 | 0.67                |
| Sample 3     | 0.8              | 0.13              | 57.16 | 6.22 | 0.66                | -                | -                 | -     | 0.69 | -                   |

Equivalent alkaline = Na<sub>2</sub>O + 0.685 K<sub>2</sub>O.

**Table 21** - Expansion of the mortar bars prepared with quartzite samples and cements CP II-Z-32 e CP V-ARI, at the age of 16 and 30 days.

| Cement     | Age     | A1     | A2     | A3     | A4     |
|------------|---------|--------|--------|--------|--------|
| CP II-Z-32 | 16 days | 0.033% | 0.031% | 0.050% | 0.116% |
|            | 30 days | 0.089% | 0.077% | 0.116% | 0.211% |
| CP V-ARI   | 16 days | 0.036% | 0.054% | 0.023% | 0.046% |
|            | 30 days | 0.194% | 0.224% | 0.108% | 0.138% |

practically identical, this difference may be attributed to the addition of pozzolana to the CP II-Z-32 cement, which accounts for the minimization of the reactions, however, in this case it was not sufficient to inhibit the expansions of A4 sample.

None of the bars tested has undergone important ruptures or fissures. Only the bars of A2 sample underwent some signs of small fissures and a trend to span.

During the casting of the mortar bars of A3 sample using the CP V-ARI cement some difficulties were checked in density increasing due to a more elevated rate of water absorption of the aggregates regarding the other samples, resulting in bigger empties in the mortar in hardened state. The irregular form of the quartzite grains accounts for this phenomenon, in this case the flat and elongated particles and angularity, which do not promote a proper compactness to concretes and mortars. The same happened in other samples, where relevant proportion of emptiness and pores in the mortar bars in the hardened state was observed.

The big quantity of empties which appeared, principally in the A3 sample specimens make that the gel coming from the reaction have more empty spaces to house, which can result in an equivocal classification of the reactive potentiality of the sample.

It was observed by Valduga *et al.* (2005) the influence of the grain form in the results of the accelerated test. The authors in this paper have verified that when the mortars are casted with more angular grains they have smaller expansions than when the aggregates have round grains, due exactly to the empties presented in those mortars, where the gel houses.

## 4.2. Results of the studies carried out in the concrete specimens

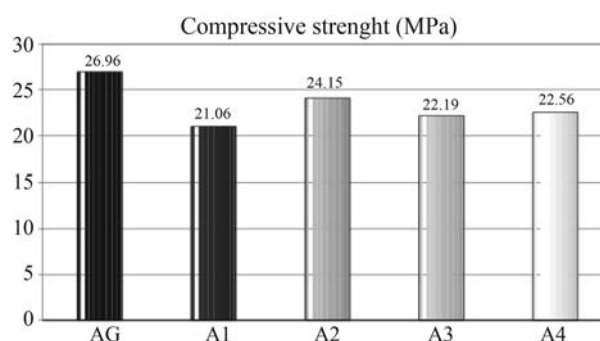
### 4.2.1. Phase 1

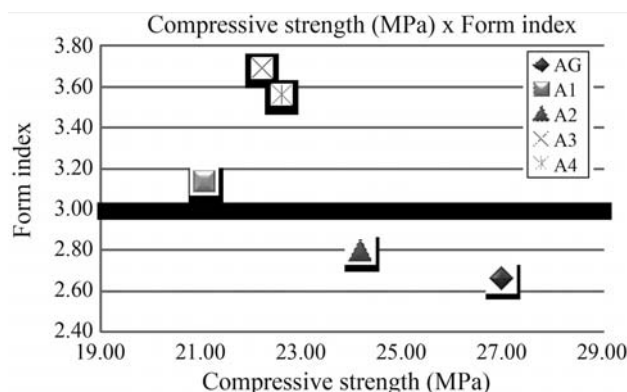
#### 4.2.1.1. Compressive strength

The results of the compressive strength obtained in 28 days in the concrete cylindrical specimens in Phase 1 are presented in Fig. 8. It is observed that the values of the strength to concrete compression produced with quartzite coarse aggregate, were smaller than the reference concrete, but they were not relevant. The maximum difference obtained was 21.88% in trace A1 in relation to AG, and the minimum difference was only 10.42% in trace A2.

Figure 9 shows a comparison between the concrete strength and the respective shape index of the coarse aggregate. The results show that, values of shape index (length x thickness ratio) lower than the limit established by ABNT (1983a), resulted in compression strengths higher than in the concretes. On the other hand, the results of the shape index which were not considered approved for the other aggregates, that is, values higher than 3.0 obtained compressive strengths lower in the concretes. The most favorable conditions of shape index approved in the tests by ABNT (1983a) are attributed to AG and A2 aggregates; the values of compressive strength of the traces produced with these two aggregates were exactly the highest and were similar.

The smallest concrete strengths produced with more lamellar grains may be attributed to the increase of the water/cement ratio in the transition zone and formation of bigger

**Figure 8** - Compressive strength of the concrete at 28 days of age (MPa).



**Figure 9** - Relation between the values of the aggregate shape index and the compressive strength (MPa) of the concretes.

crystalline grains reflecting in bigger porosity in aggregate interface and paste and consequently, region weakening.

After the tests of compressive strength the concretes were taken to a complete rupture so that the rupture section could be analyzed. The intention was to verify, through the visual analysis, whether the concrete rupture was happening in coarse aggregate or in cement paste. Figure 10 presents images of sections of rupture of specimens.

Analyzing the specimens produced with coarse gneiss and quartzite aggregates, it was verified that there were spoiled aggregates and also aggregates taken from the paste (paste rupture around the aggregate), which characterizes the proper aggregate adherence to the paste. It was also observed that the quantity of spoiled aggregates was smaller or approximately the same as the aggregates separated from the cement paste, indicating that the aggregate strength is also acceptable. It was also observed in all concrete traces that most of the particles were wrapped with cement paste in sections of rupture.

#### 4.2.1.2. Tensile Strength by diametral compression

The results of tensile strength by diametral compression at the age of 28 days are presented in Fig. 11. The val-

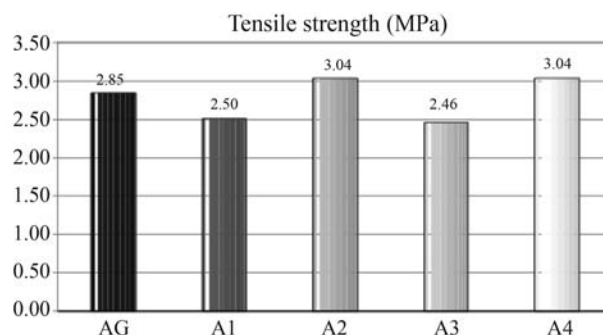
ues presented about the tensile strength were satisfactory with no relevant difference between the samples. A1 and A3 traces obtained inferior values in relation to AG trace in 12.5% whereas A2 and A4 obtained a tensile strength increase in relation to AG trace in 6.28%.

According to Mehta & Monteiro (2008) the concrete strength, specially the flexural strength, may be affected by the aggregate texture. Rougher textures may promote the formation of a stronger physical adherence between the cement and the aggregate. The porosity rate is one of the characteristics which account for a rougher texture to the coarse aggregate.

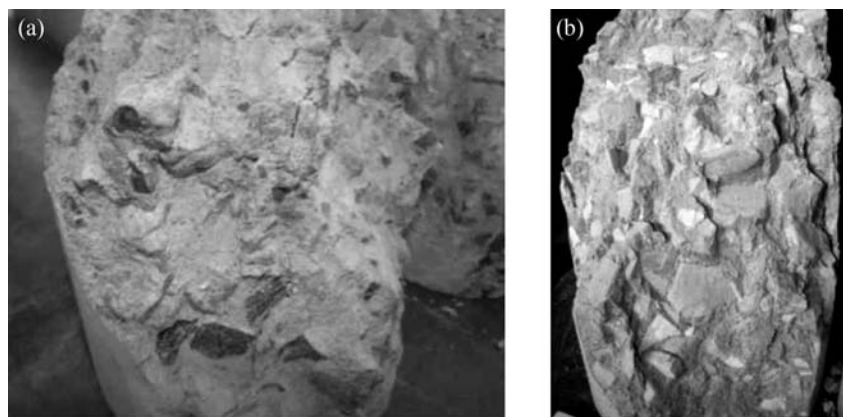
Other factors which are dependent to classify a superficial texture, mentioned by Metha & Monteiro (2008), can be attributed to petrographic origin, hardness, grain size and exposition to friction. Fig. 12 presents a rupture section of some specimen tested on tension by diametral compression.

Studies conducted by Tasong *et al.* (1998a) verified that the strength linking the concrete interface produced with quartzite with sawn surfaces was higher when compared to basalts and limestone with the same kind of surface.

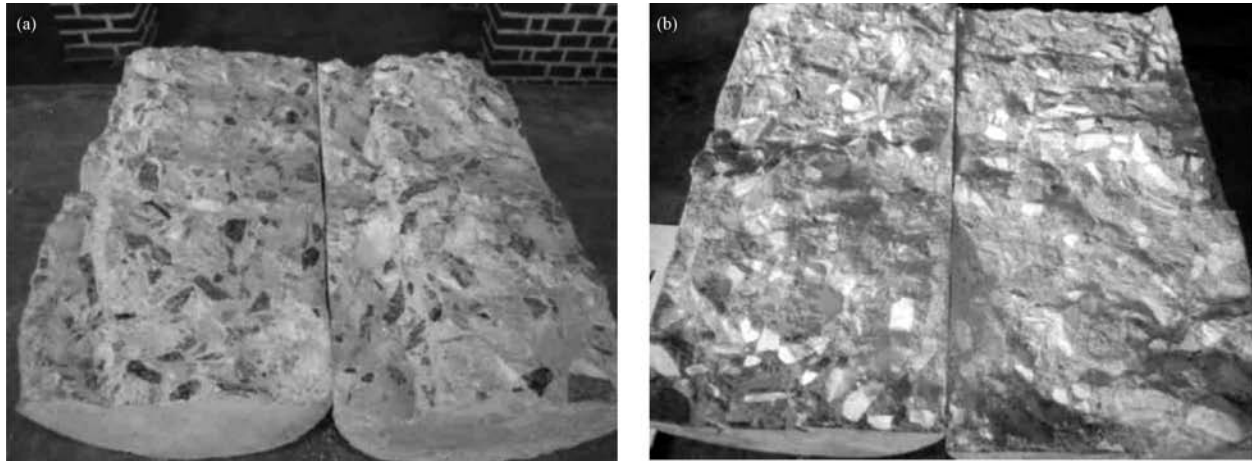
The authors believe that the pozzolanic reactions between the quartzite Si and the CH may be the reason for the high strength in the linking. The authors suggest perform-



**Figure 11** - Tensile strength by diametral compression of aggregates on the 28<sup>th</sup> day.



**Figure 10** - Sections of rupture to the compression of concretes produced with the traces AG(a) and A1(b).



**Figure 12** - Rupture sections of specimens corresponding to AG(a) and A1(b) traces.

ing not only analysis of interaction between aggregate and cement paste in the concrete transition zone, but fundamentally analysis of the behavior of the superficial aggregate texture and the kind of fissure which occurred in the transition zone, being considered interfering factors confirmed in their research.

In investigations using powder-like quartzite in cement-based solutions, Tasong *et al.* (1998b) proved that the quartzite is much more active than expected. During the test the quartzite absorbed relevant quantities of OH and Ca and released relevant quantities of Si for the solution, this point allows the indication of the formation C-S-H on the aggregate surface.

#### 4.2.2. Phase 2

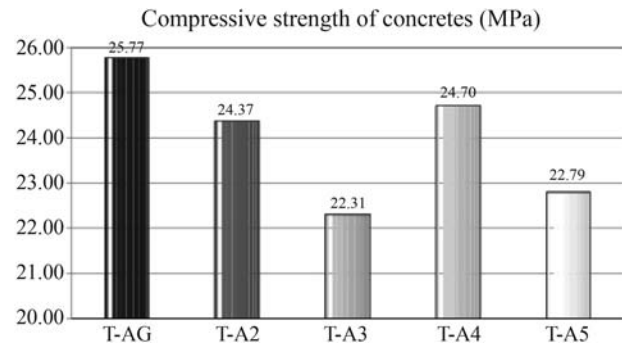
##### 4.2.2.1. Compressive strength

Figure 13 shows the maximum values of compressive strength obtained through specimens tested at the age of 28 days.

The value differences of the concrete compressive strength were very close, showing to be on a par with the concretes produced with quartzite aggregates in relation to the reference concrete produced with gneiss aggregate. The maximum difference obtained was 13.43% in T-A3 trace in relation to T-AG and the minimum difference was only 4.16% in T-A4 trace.

It is important to point out that in Phase 1 the differences between the concretes were bigger and attributed to the irregular forms of the aggregate fragments, however in this phase these deficiencies were corrected with the help of the hydrocone crusher reflecting in better mechanic properties concretes.

After the tests of compressive strength, the concretes were taken to complete rupture so that the rupture section could be analyzed. The intention was to verify through visual analysis whether the rupture of the concretes was occurring in the coarse aggregates or in the cement paste.



**Figure 13** - Compressive strength of concretes at the age of 28 days.

Figures 14(a) and 14(b) present images of the rupture sections of the specimens.

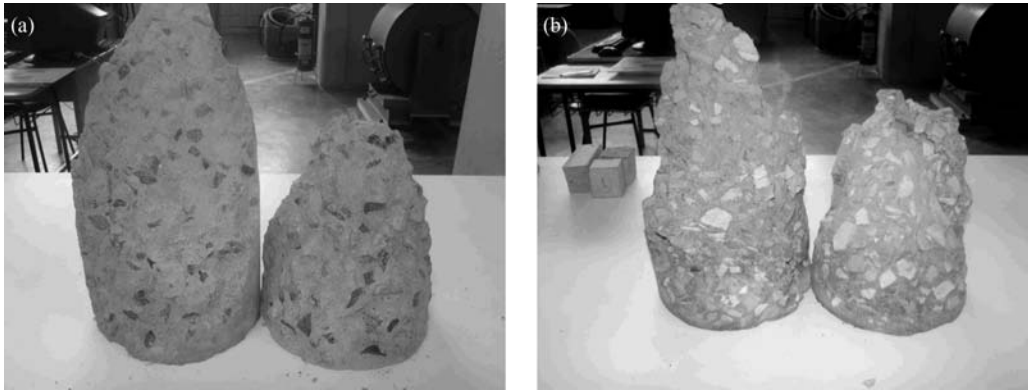
Just like in Phase 1 it was possible to verify that there were, in the rupture region of specimens, spoiled aggregates and also aggregates taken from the paste (rupture from the paste around the aggregate), which characterizes the proper adherence of the aggregate to the paste.

It was also possible to observe that the quantity of spoiled aggregates was smaller, or approximately equal to the aggregates separated from the cement paste indicating that the aggregate strength is also acceptable. It was observed in all concrete traces that most particles were wrapped with the cement paste in rupture sections.

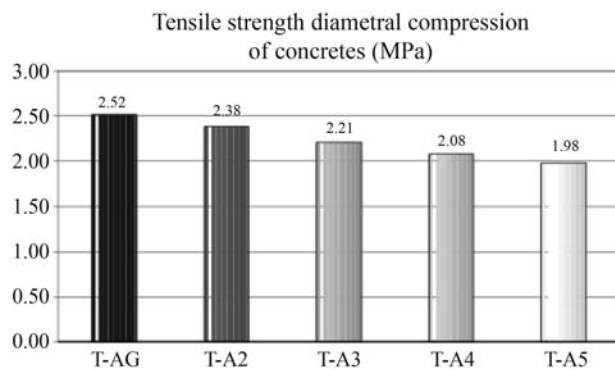
##### 4.2.2.2. Tensile strength by diametral compression

Figure 15 shows the results of tensile strength by diametral compression at the age of 28 days.

The values presented of tensile strength were satisfactory, not representing relevant differences with the samples. The biggest difference was 21.43% in T-A5 trace in relation to T-AG and the smallest difference was only 5.55% in relation to T-AG trace.



**Figure 14** - Rupture sections to compression of concretes produced, corresponding to AG(a) and A4(b).



**Figure 15** - Tensile strength by diametral compression of concretes at the age of 28 days.

Figure 16 presents the rupture section of the specimens tested under tension by diametral compression.

#### 4.2.2.3. Modulus of elasticity

The results of the tests of the modulus of elasticity carried out in Phase 2 are shown in Table 22.

The biggest difference obtained was 7.67 GPa in trace T-A3 in relation to T-AG. And the smallest difference was only 5.04 GPa in trace T-A2 in relation to T-AG.

**Table 22** - Results of the modulus of elasticity.

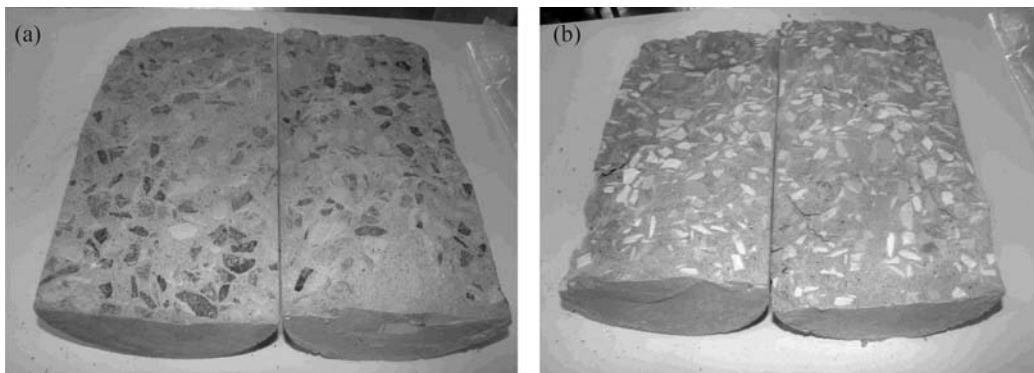
| Samples | $f_{cj}$ (MPa) | % $f_{cj}$ | ME (GPa) |
|---------|----------------|------------|----------|
| T-AG    | 23             | 20         | 21.58    |
| T-A2    | 20             | 20         | 16.54    |
| T-A3    | 20             | 20         | 13.91    |
| T-A4    | 21             | 20         | 15.42    |
| T-A5    | 18             | 20         | 16.09    |

The values (results) were very close when it comes to quartzite samples, but when they are compared to the reference trace this variation is considerable. The difference may be attributed, for example, the interaction between aggregate and binder paste in the transition zone.

It is important to point out that microscopic studies can help interpret these differences of modulus of elasticity and that specific dosage studies can make the performance of the concrete modulus of elasticity better, through variations of the a/c ratio, reduction, coarse aggregate content, among others.

## 5. Conclusion

Two distinct lithological kinds of waste from quartzite mines in the region were identified: Type 1- which is



**Figure 16** - Rupture sections of the specimens corresponding to the sample traces AG(a) and A5(b).

constituted by the remaining foliate quartzite, mica - which is effectively used as “coating stone”; and Type 2, which is a silicified quartzite, with low mica content, non-foliated and that after the extraction, it is completely discharged. Type 2 due to its physical, mineralogical, and mechanical characteristics was selected in this study.

The results to characterize the quartzite aggregates, in general, were quite satisfactory compared to the conventional gneissic aggregate, to the rates presented by Verhoef e Van De Wall (1998 *apud* Gomes, 2001) and to the parameters established by ABNT (2005).

The deficiencies of shape index found in aggregates in Phase 1 using the jaw crusher according to ABNT (1983a) were corrected in Phase 2 using the hydrocone crusher.

Regarding the alkali-aggregate reactivity done by ASTM (2007) accelerated test, the deleterious potentiality was verified both, with CP II-Z-32 and CP V-ARI cements. In this case it is recommended the use of low-content alkali and with additions such as the CP III (High-Furnace Portland Cement) and the CP IV (Pozzolanic Portland Cement) and preventive methods of the reaction such as the water-proofing structure, for instance. However, further studies using different methods should be carried out for a final conclusion of the quartzite reactivity.

The results of the properties in hardened state were also satisfactory. The differences obtained in Phase 1 after the compressive strength test (maximum 21.88% in A1 and minimum 10.42% in A2 in relation to AG) were attributed to the irregular forms of the grains affecting directly the concrete strength. The bigger the proportion of flat and elongated particles, the bigger the trend for accumulation of water film along the aggregate surface will be, weakening the transition zone in the aggregate-paste interface.

In Phase 2, the differences obtained after the compression test (maximum 13.43% in T-A3 trace and minimum 4.16% in A4 trace in relation to the T-AG concrete) showed the efficacy of the hydrocone crusher demonstrating that the results between the quartzite and gneiss concretes were very close.

The results of the modulus of elasticity, carried out in Phase 2 presented considerable variations between the quartzite and gneiss traces, however, they were next to the conventional interval of the concretes to use in structures which is 16 GPa to 35 GPa. Thus, alternative dosage studies varying the characteristics of concretes and analyses of the internal microstructure of these concretes are recommended for a better interpretation of these differences.

In Phase 1 it was observed visually that not all the aggregate grains presented a formation of a cement paste layer on its surface. The arrangement of the aggregate particles inside the concrete is also an interfering factor, due to the flat and elongated forms of some grains, which make it difficult the compactness and homogeneousness of the material. In Phase 2 the grains presented greater adherence to

the cement paste, confirming the efficacy of the quartzite aggregates when processed in proper crusher.

In a last analysis, the studies carried out in this paper indicated that the quartzite residues coming from mines in the Southwestern Minas Gerais presented satisfactory behavior for the use as a coarse aggregate in the concrete.

With further research and also the support of institutions that contribute to the moving of regional economy, the commercialization of this product will be feasible and it will also contribute to solve social, economical, and environmental problems which reach the mining sector of the Southwest of the state, especially when it comes to quartzite.

## 6. Recommendations

As recommendations for future studies, it is suggested:

- Study concretes with different consistency and strength;
- Carry out studies of potential alkali reactivity of aggregates, using alternative methods, other kinds of cement and check the interference of the grain form on the empty index of mortars;
- Analyze the microstructure of concretes to check the aggregate interface and binder paste, principally in more advanced ages.

## Acknowledgments

We thank FAPEMIG and Fundação de Ensino Superior de Passos (FESPUEMG) for financial and logistical support to this research and trainees Eridano Valim dos Santos Maia, Guilherme Alves Cândido and Morrâmulo Ítalo Pereira Granja for the support.

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# Characterization, Hydraulic Conductivity and Compatibility of Mixtures of Tropical Soil and Bentonite for Barrier Usage Purpose

T.L.C. Morandini, A.L. Leite

**Abstract.** Compacted tropical soils have great potential to be used in barriers, once some technical issues are correctly addressed, including low hydraulic conductivity and compatibility to the disposed fluids. This paper involves a laboratorial study of the hydraulic conductivity of mixtures of a tropical soil sample and bentonite and also their compatibility to different chemical solutions. The experimental program consisted of (1) sample characterization, (2) hydraulic conductivity determination using a triaxial cell, and (3) compatibility assessment with different chemical solutions ( $\text{HNO}_3$ ,  $\text{NaOH}$ ,  $\text{NaCl}$ , and ethanol) using the modified Atterberg Limits. The characterization results revealed important changes in the index properties of the tropical soil sample as the result of the bentonite addition, especially in the plasticity, cation exchange capacity and free swelling. The hydraulic conductivity, in turn, experimented significant decreasing as the proportions of bentonite and confining pressure increased. The compatibility results showed a significant reduction in the plasticity of the samples when subjected to the chemical solutions, especially for the salt solution. In general terms, the addition of bentonite reduced the compatibility of the samples.

**Keywords:** tropical soils, bentonite, hydraulic conductivity, compatibility, compacted clay barriers.

## 1. Introduction

Compacted clay barriers are widely used for waste containment throughout the world, including domestic, industrial, health-care and radioactive refuse. Base liners, also called compacted clay liners (CCL), are intended to isolate the waste from the nearby environment, especially groundwater. Additionally, cover systems of compacted clay are designed to reduce the rain infiltration, decreasing the leachate generation inside the refuse (Rowe, 2001; Farnezi & Leite, 2007; Chalermyanont *et al.*, 2009).

A CCL mainly works by reducing the hydraulic conductivity ( $k$ ) and by retarding the migration of contaminant through sorption mechanisms (Daniel & Benson, 1990; Benson *et al.*, 1994; Allen, 2001; Egloffstein, 2001; Kominé, 2004). So, suitable materials for CCL construction must fulfill technical requirements mainly represented by low  $k$  values,  $10^{-9}$  m/s as an example, and high sorption capacity. Other issues play also an important role, such as low compressibility and long term performance. In addition, the maintenance of the barrier properties when in contact with the waste liquids, called compatibility, should be considered (Eklund, 1985; Bouazza, 2002).

Due to their wide distribution over the globe, tropical soils have a great potential to be used in these compacted barriers, once some technical and economical requirements are satisfied. The origin of tropical soils is related to hot and humid climates, and vast extensions of these soils cover some old plateaus of South America, Africa and other tropi-

cal parts of the world. Their occurrence is characterized by thick and relative homogeneous weathering profiles as the result of advanced weathering. Their typical composition is quite peculiar, comprising quartz, Fe-Al oxides/hydroxides and kaolinite clay, which makes them not as active as most of the soils of cold and temperate climates.

Additionally, some authors such as Pandiam *et al.* (1993), Cozzolino & Villibor (1993) and Fernandes (2005) consider that some conventional geotechnical soil classification systems do not give satisfactory results for tropical soils. They attribute this discrepancy to some specific properties of the tropical soils such as: 1) soil aggregation, which may exert influence on the grain size analysis and Atterberg Limits; 2) surface chemistry: alkaline conditions may originate positive charges on the solid surface, which increases the interfacial tension and capillarity; 3) silt plasticity, originated by large crystals of kaolinite and mica and 4) pore configuration: some soils from tropical climates exhibit large voids as the result of flocculation and grain fragmentation, which may alter grain size distribution and soil saturation conditions.

In spite of their extensive occurrence, sometimes tropical soils do not attend the low  $k$  conditions required for barrier construction, because of its aggregate nature. Cozzolino & Villibor (1993) mention the aggregate (soil peds) and porous nature of tropical soils, resulting in high hydraulic conductivities. Besides, due to their low activity, the potential of retarding contaminants is restricted.

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Submitted on March 27, 2012; Final Acceptance on November 21, 2012; Discussion open until April 30, 2013.

Bentonite clays are well known by their large swelling, high cation exchange capacity (CEC) and sorption capacity, high plasticity and low hydraulic conductivity (Laird, 2006; Jan *et al.*, 2007). Thus, bentonite can be a potential additive to reduce the permeability and increase the sorption capacity of tropical soils. The results presented by Anderson & Hee (1995), Osinubi & Nwaiwu (2002), Farnezi & Leite (2007), Nayak *et al.* (2007) and Sunil *et al.* (2009) confirm this potentiality.

Some papers have assessed the addition of bentonite to natural sands for reducing the hydraulic conductivity, such as Chapuis (1990), Keeney *et al.* (1992), Sivapullaiah *et al.* (1998), Stewart *et al.* (2003), Ebina (2004) and Kominé (2004). Other papers describe laboratory studies to investigate the possible changes in the properties of soils from cold and temperate climate when exposed to different chemical solutions (Bowders & Daniel, 1987; Madsen & Mitchell, 1989; Budhu *et al.*, 1990; Shackelford, 1994; Shackelford *et al.*, 2000; Jo *et al.*, 2001; Roque & Didier, 2006; Yamaguchi *et al.*, 2007; Castellanos *et al.*, 2008; Katsumi *et al.*, 2008; Smiles, 2008; Cuisinier *et al.*, 2009; Kinsela *et al.*, 2010). Therefore, it is latent the need of a more specific research on the hydraulic properties of tropical soils and their long term performance (compatibility) to different liquids.

This paper investigates a tropical soil sample and its mixtures with bentonite in the proportions of (dry weight basis) 3, 6, 9 and 12% of this additive. The experimental program was mainly intend to elucidate the following issues: (1) the actual effect of the bentonite addition to the geotechnical and physico-chemical properties of the tropical soil sample, which was achieved by an extensive characterization; (2) the possible reduction in the  $k$  value, assessed through permeability tests using a flexible wall permeameter (triaxial cell) and (3) the compatibility of the soil samples when exposed to different chemical solutions ( $\text{HNO}_3$ ,  $\text{NaOH}$ ,  $\text{NaCl}$  e ethanol) by means of the evaluation of modified Atterberg limits and modified free-swelling test.

## 2. Sample Preparation

The bentonite sample used in the tests is commercially called BRASGEL, and according to the manufacturer it came from the state of Paraíba, northeastern Brazil. In turn, the tropical soil sample was collected on a soil outcrop by the road that connects the cities of Mariana and Ponte Nova, in the state of Minas Gerais, southeastern Brazil. In the field, the reddish weathering profile was quite thick and homogeneous. Soil peds and blocky structure dominate the outcrop.

The proportions of bentonite used to compose the mixtures were defined according to previous work, such as Anderson & Hee (1995); Gardner & Arias (2000); Ryan & Day (2002); Farnezi & Leite, (2007); Jan *et al.* (2007). In

this way, the following denominations and proportions are given (dry weight basis):

- SN: 100% natural soil;
- SN03: 97% natural soil + 3% bentonite;
- SN06: 94% natural soil + 6% bentonite;
- SN09: 91% natural soil + 9% bentonite;
- SN12: 88% natural soil + 12% bentonite;
- BB: 100% bentonite.

The sample preparation for the characterization and compatibility tests was guided by the Brazilian standard NBR-6457 (ABNT, 1986). The samples were firstly dried at room temperature, and then homogenized, sieved and reduced. The samples used in the hydraulic conductivity tests were compacted under Proctor Energy as indicated by NBR-7182 (ABNT, 1986), with the moisture content around 2% above the optimum. After compaction they were cut and trimmed to a diameter of 5 cm and 10 cm height.

## 3. Sample Characterization

### 3.1. Procedures

Physico-chemical properties (pH, electrical conductivity of aqueous extract, cation exchange capacity and specific surface) and geotechnical properties (grain size distribution, liquid limit, plastic limit, specific gravity of solids, optimum moisture content and maximum dry unit weight) were determined for the natural soil, bentonite and mixtures, following the references listed in the Table 1.

According to Camargo *et al.* (1986), the sample pH can be determined in a suspension of 1:2.5 soil/solution ratio, and the electrical conductivity (EC) is measured in the water extract of a suspension of 1:1 soil/solution ratio. The CEC and specific surface (SS) of the fraction passed through the 2 mm sieve were estimated using the method of blue methylene adsorption on a filter paper (Pejon, 1992).

**Table 1** - References used for sample characterization.

| Properties                                                                                                                  | References                   |
|-----------------------------------------------------------------------------------------------------------------------------|------------------------------|
| Grain size distribution                                                                                                     | ABNT-NBR7181 (1984)          |
| Liquid limit ( $\omega_L$ )                                                                                                 | ABNT-NBR6459 (1984)          |
| Plastic limit ( $\omega_p$ )                                                                                                | ABNT-NBR7180 (1984)          |
| Specific gravity of solids ( $G_s$ )                                                                                        | ABNT-NBR6508 (1984)          |
| Optimum moisture content ( $w_{ot}$ ) and maximum dry unit weight ( $\rho_{dmax}$ )                                         | ABNT-NBR7182 (1986)          |
| pH determination in $\text{H}_2\text{O}$ ( $\text{pH}_{\text{H}_2\text{O}}$ ) and KCl solution ( $\text{pH}_{\text{KCl}}$ ) | Camargo <i>et al.</i> (1986) |
| Electrical conductivity of aqueous extract (EC)                                                                             | Camargo <i>et al.</i> (1986) |
| Cation exchange capacity (CEC) and specific surface (SS)                                                                    | Pejon (1992)                 |

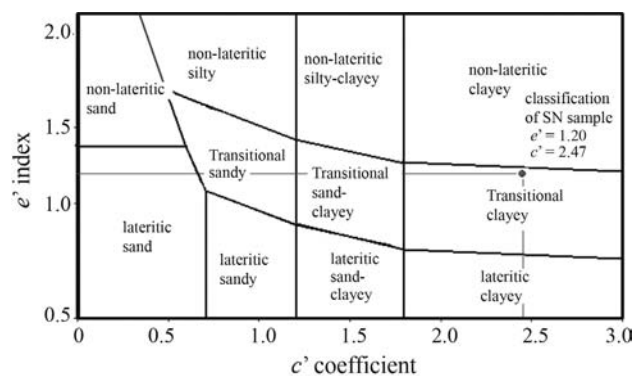
X-ray diffraction was used to determine the mineralogy of the samples that passed through the 0.075 mm sieve under the conditions of: copper tube; rotation angles of 2 to 70° and velocity of rotation from 1 to 2° per second. For that, oriented thin sections were confectioned and submitted to ethylene glycol solvation and heating to 550 °C for 4 h.

The lateritic character of the tropical soil sample (SN) was investigated using a Modified MCT Classification (Vertamatti, 1988). For that, the Brazilian standards DNER-M256-94 and DER-M196-89 were applied.

### 3.2. Results

As can be seen in Fig. 1, the SN sample was classified as *transitional clayey* in the modified MCT classification system (Vertamatti, 1988), which refers to a transitional field between lateritic and non-lateritic. On the other hand, the outcrop conditions in which the sample was collected, characterized by a dark reddish color and deep/homogeneous weathering profile, clearly indicates that laterization is occurring.

The grain size distribution of all the samples is presented in Table 2. It is interesting to notice that the addition of bentonite had no influence on the clay fraction of the SN sample, despite its high content of clay (see BB sample in



**Figure 1** - Modified MCT classification of the SN sample (abacus as Vertamatti, 1988).

**Table 2** - Grain size distribution of the samples.

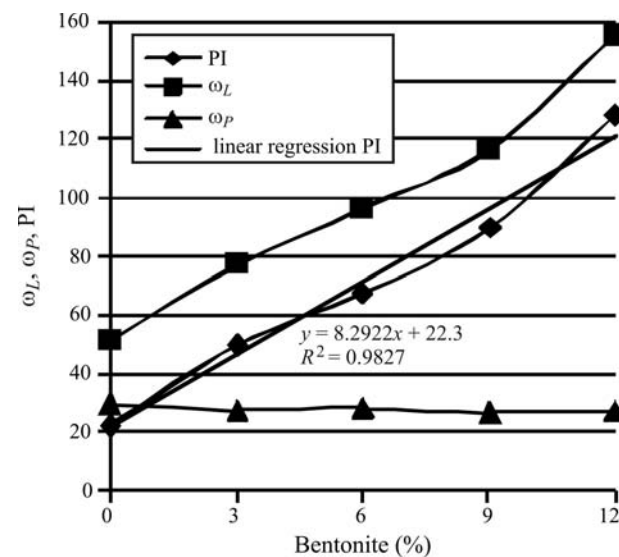
| Grain-size analysis <sup>1</sup> | Sample |      |      |      |      |    |
|----------------------------------|--------|------|------|------|------|----|
|                                  | SN     | SN03 | SN06 | SN09 | SN12 | BB |
| Clay (%)                         | 42     | 42   | 42   | 42   | 42   | 91 |
| Silt (%)                         | 6      | 3    | 6    | 6    | 6    | 9  |
| Fine sand (%)                    | 6      | 13   | 10   | 8    | 10   | 0  |
| Medium sand (%)                  | 36     | 30   | 32   | 32   | 32   | 0  |
| Coarse sand (%)                  | 8      | 10   | 8    | 10   | 8    | 0  |
| Gravel (%)                       | 2      | 2    | 2    | 2    | 2    | 0  |

<sup>1</sup>Grain-size scale of the Massachusetts Institute of Technology.

Table 2). It is supposed that addition of bentonite has promoted the aggregation or flocculation of the silt and sand grains size of the natural soil (SN sample), which “increases” the “size” of the grains even under the influence of the deflocculating agent (sodium hexametaphosphate, 45.7 g/L). This aggregation could be noticed by a visual inspection in the laboratory.

Some other properties are presented in the Table 3. As was expected, the BB sample showed high values of plasticity, activity, pH, *EC*, *CEC* and *SS*. In turn, the SN sample presented acidic pH and a  $\Delta$ pH around zero (slightly positive), which is quite common for tropical soils.

The influence of the bentonite addition on the Atterberg Limits, *CEC* and *SS* is demonstrated on Figs. 2 and 3, respectively. Figure 2 shows that the increase in the Plastic Index with the bentonite content was quasi-linear ( $R^2 = 0.983$ ), mainly influenced by the increase in the Liquid Limit. *CEC* and *SS*, in turn, experimented a significant increase with the bentonite addition (Fig. 3).



**Figure 2** - Atterberg limits ( $\omega_L$ ,  $\omega_P$ , *PI*) with bentonite addition.

Figure 4 shows the compaction curves for all the samples. It is clear the influence of the bentonite addition on the compaction behavior, increasing the  $w_{ot}$  and decreasing  $\rho_{dmax}$ .

The x-ray diffraction analysis of the SN sample has showed a mineralogy predominantly composed of kaolinite, gibbsite, hematite and goethite, which is typical for tropical soils. Additionally, the BB sample was mainly composed of smectite, with small amounts of kaolinite, quartz and mica, as shows Figs. 5 and 6.

The high content of aluminum ( $Al_2O_3 = 15.4\%$ ) and iron ( $Fe_2O_3 = 26.7\%$ ) of the SN sample indicates that extensive leaching (laterization) took place in the soil formation. On the other hand, the percentage of silica ( $39.2\%$ ) is transitory between lateritic and non-lateritic soils.

## 4. Hydraulic Conductivity

### 4.1. Procedures

The hydraulic conductivity ( $k$ ) of the samples was determined using a flexible wall permeameter (triaxial cell). The test methods were based on the suggestions of Head (1986), involving backpressure saturation and pre-consolidation of the samples of 5 cm in diameter and 10 cm high. Sample saturation was obtained by backpressure to a limit of 300 kPa and saturation was considered fulfilled when the  $B$  parameter reached a minimum of 0.94. After these procedures are completed, samples were percolated under constant head of 50 kPa.

Hydraulic conductivity testing was not performed in the bentonite due to the difficulties faced in compacting, shaping and saturating this sample.

The schematic of the apparatus used in the tests is depicted on Fig. 7. Pressure application systems were installed at the base ( $p_1$ ) and over the top ( $p_2$ ) of the soil specimens, along with the confining pressure system ( $\sigma_3$ ). A transducer for pore pressure ( $u$ ) monitoring and an electronic gage for flow measures were coupled to the triaxial cell. Anderson and Hee (1995), Shackelford *et al.* (2000), and Ahn & Jo (2009) have obtained satisfactory results with similar equipment.

The consolidation effective stresses of 20, 40 to 80 kPa have been applied as an attempt to simulate field pressures on CCL induced by municipal solid waste (MSW). The void ratio ( $e$ ) during consolidation was estimated through the Eq. 1.

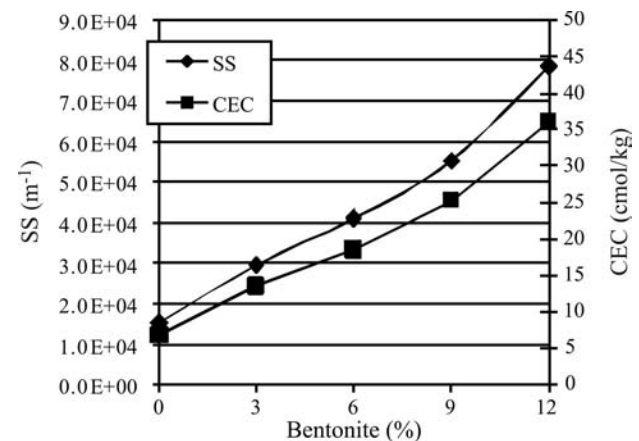
$$e = e_0 - (1 + e_0) \frac{\Delta V}{V_0} \quad (1)$$

where  $\Delta V$  is the volume variation of the sample,  $V_0$  is its initial volume and  $e_0$  is its initial void ratio.

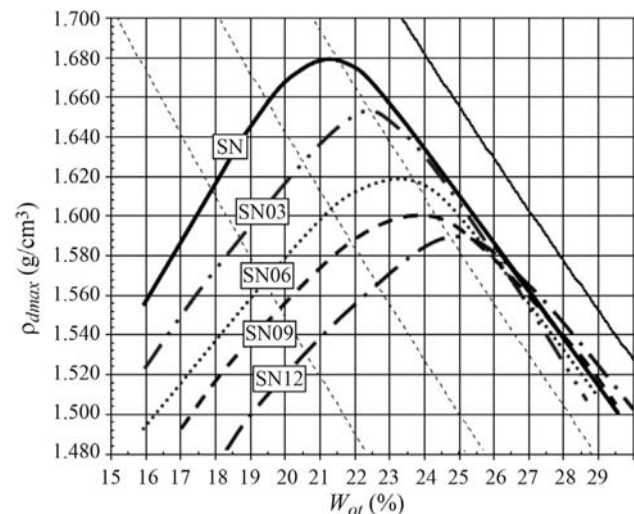
Flow was imposed to the soil specimen by the application of a  $p_1$  of 300 kPa, while  $p_2$  was kept constant at 250 kPa. These conditions have produced a hydraulic head of 50 kPa and a hydraulic gradient ( $i$ ) in the order of 50. It is

**Table 3** - SN and BB sample properties.

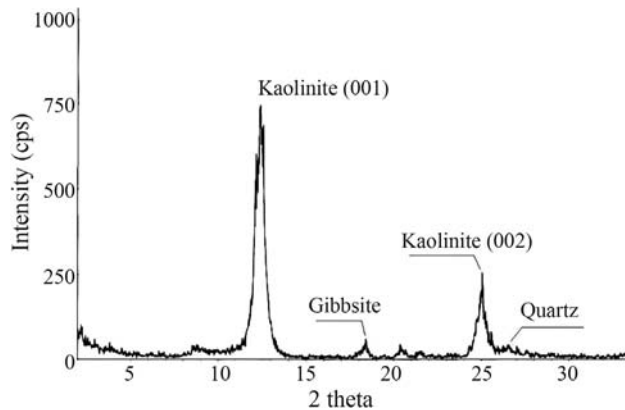
| Property                                                | Sample             |                    |
|---------------------------------------------------------|--------------------|--------------------|
|                                                         | SN                 | BB                 |
| Liquid Limit - $\omega_L$ (%)                           | 51.9               | 682.5              |
| Plastic Limit - $\omega_p$ (%)                          | 29.6               | 90.6               |
| Plasticity Index - $PI$ (%)                             | 22.3               | 591.9              |
| Activity                                                | 0.53               | 6.50               |
| Specific gravity of solids - $G_s$ (Mg/m <sup>3</sup> ) | 2.840              | 2.452              |
| pH in H <sub>2</sub> O - $pH_{H_2O}$                    | 5.15               | 9.92               |
| pH in KCl solution - $pH_{KCl}$                         | 5.21               | 8.77               |
| $\Delta pH = pH_{KCl} - pH_{H_2O}$                      | 0.06               | -1.15              |
| Electrical conductivity - $EC$ (mS/cm)                  | 0.05               | 1.30               |
| Cation Exchange Capacity - $CEC$ (cmol/kg)              | 6.9                | 99.4               |
| Specific Surface - $SS$ (m <sup>-1</sup> )              | $1.53 \times 10^4$ | $1.90 \times 10^5$ |



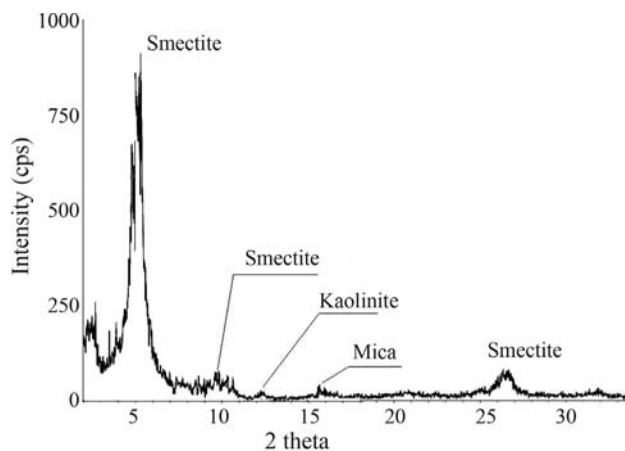
**Figure 3** - Specific surface ( $SS$ ) and Cation Exchange Capacity ( $CEC$ ) of the SN sample with bentonite addition.



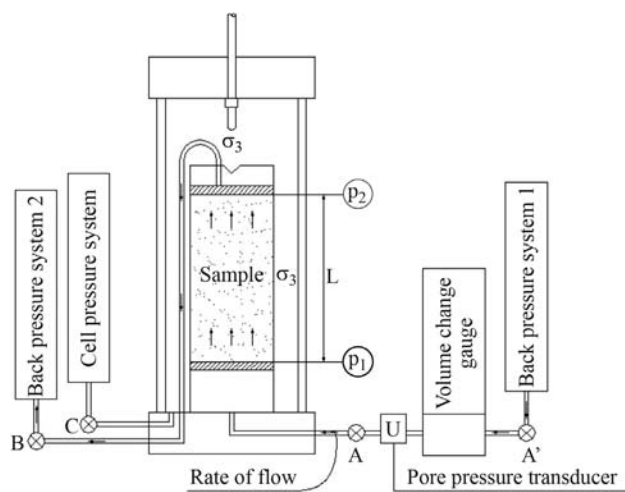
**Figure 4** - Compaction curves of all the samples.



**Figure 5** - X-ray diffraction analysis of the SN sample.



**Figure 6** - X-ray diffraction analysis of the BB sample.



**Figure 7** - Schematic of the apparatus used in the hydraulic conductivity tests.

recognized that this gradient is very high when compared to ordinary field conditions (gradients of 3 or less), however it was necessary for operational reasons, since very low hydraulic conductivities are expected for compacted clay samples.

Equation 2 (Darcy Law) was used for  $k$  [m/s] calculation when the flow conditions were stable. This stability was considered when the discharge,  $Q$  [m<sup>3</sup>], was linear over the time,  $t$  [s].

$$k = \frac{Q}{A \Delta t} = K \frac{\gamma}{\mu} \quad (2)$$

where  $A$  is the soil cross sectional area,  $K$  [L<sup>2</sup>] the soil intrinsic permeability,  $\gamma$  [ML<sup>-3</sup>] the fluid unit weight and  $\mu$  [ML<sup>-1</sup>T<sup>-2</sup>] the fluid dynamic viscosity.

Some aspects of the tests are pointed out: (1) because of operational reasons - difficulties in the compaction, molding, saturation and consolidation, the hydraulic conductivity of the BB sample was not measured. Anyway, all the characteristics of this sample illustrated on Figs. 2 and 3 (high clay content, high plasticity, high activity etc) have lead to the conclusion that it really is a quasi impermeable material; (2) porous stones placed at the base and top of the soil specimen prevented fine percolation; (3) the rule  $\sigma_3 > p_1 > p_2$  has been obeyed to avoid the sample liquefaction induced by pore pressure increase; (4) the reported  $k$  results (see item 4.2) is an arithmetic average of 3 measures.

## 4.2. Results

All the hydraulic conductivity results are presented in Table 4. In this table  $k_i$  refers to the  $k$  value of each soil specimen under different confining pressure,  $k_a$  refers to the arithmetic average of the three measures, and the *Dif.k* parameter represents a percentage difference of the  $k_i$  value relative to the average  $k_a$  according to the Eq. 3.

$$Dif.k (\%) = \frac{k_i - k_a}{k_a} \times 100 \quad (3)$$

It can be noticed that the *Dif.k* parameter increases towards the highest contents of bentonite. This may be a consequence of the reduction of  $k$ , once the difficulty to measure very small flow volumes increases.

The dispersion of the *Dif.k* parameter can be evaluated by the standard deviation and the average deviation presented in Table 5. At first sight, these values seem to be quite high, with standard deviation of more than 14%. Considering the natural inaccuracy involved in the determination of  $k$  in laboratory, this dispersion can be viewed as ordinary. Once more the influence of the bentonite is apparent, since the greatest dispersions occur for the samples with more bentonite.

The reduction of the  $k$  values with the bentonite addition under increasing confining pressure is better demonstrated by the Fig. 8 and Table 6. This reduction was more expressive for the lower bentonite contents and for the higher values of  $\sigma_3$ . This trend is corroborated by the  $k_d/k_{SN}$  index presented on Table 6.

As mentioned in the literature, the value of  $k = 10^{-7}$  cm/s attend most of the regulations over the world as the

**Table 4** - Hydraulic conductivity results and the parameter *Dif. k*.

| Sample | Parameter        | $\sigma_3 = 20$ kPa |          |          | $\sigma_3 = 40$ kPa |          |          | $\sigma_3 = 80$ kPa |          |          |
|--------|------------------|---------------------|----------|----------|---------------------|----------|----------|---------------------|----------|----------|
|        |                  | Test 1              | Test 2   | Test 3   | Test 1              | Test 2   | Test 3   | Test 1              | Test 2   | Test 3   |
| SN     | $k_i$ (cm/s)     | 1.61E-06            | 1.79E-06 | 1.82E-06 | 1.07E-06            | 1.14E-06 | 1.24E-06 | 8.24E-07            | 8.02E-07 | 7.96E-07 |
|        | $k_a$ (cm/s)     |                     | 1.74E-06 |          |                     | 1.15E-06 |          |                     | 8.07E-07 |          |
|        | <i>Dif.k</i> (%) | -7.59%              | 3.17%    | 4.42%    | -6.73%              | -0.71%   | 7.44%    | 2.09%               | -0.66%   | -1.42%   |
| SN03   | $k_i$ (cm/s)     | 2.1E-07             | 1.8E-07  | 1.7E-07  | 1.3E-07             | 1.3E-07  | 1.2E-07  | 7.6E-08             | 7.4E-08  | 6.2E-08  |
|        | $k_a$ (cm/s)     |                     | 1.8E-07  |          |                     | 1.2E-07  |          |                     | 7.0E-08  |          |
|        | <i>Dif.k</i> (%) | 12.48%              | -2.21%   | -10.27%  | 0.95%               | 4.27%    | -5.22%   | 7.16%               | 4.81%    | -11.97%  |
| SN06   | $k_i$ (cm/s)     | 3.9E-08             | 4.5E-08  | 3.3E-08  | 2.1E-08             | 2.5E-08  | 1.9E-08  | 1.1E-08             | 1.3E-08  | 9.4E-09  |
|        | $k_a$ (cm/s)     |                     | 3.9E-08  |          |                     | 2.1E-08  |          |                     | 1.1E-08  |          |
|        | <i>Dif.k</i> (%) | -0.81%              | 14.73%   | -13.92%  | -2.78%              | 15.86%   | -13.08%  | 0.13%               | 14.24%   | -14.37%  |
| SN09   | $k_i$ (cm/s)     | 1.4E-08             | 1.6E-08  | 2.0E-08  | 9.5E-09             | 7.3E-09  | 9.0E-09  | 2.8E-09             | 3.5E-09  | 2.9E-09  |
|        | $k_a$ (cm/s)     |                     | 1.7E-08  |          |                     | 8.6E-09  |          |                     | 3.0E-09  |          |
|        | <i>Dif.k</i> (%) | -16.41%             | -2.95%   | 19.36%   | 10.75%              | -15.34%  | 4.59%    | -8.15%              | 14.16%   | -6.01%   |
| SN12   | $k_i$ (cm/s)     | 9.7E-09             | 1.1E-08  | 1.3E-08  | 4.7E-09             | 3.3E-09  | 4.9E-09  | 1.1E-09             | 1.4E-09  | 1.2E-09  |
|        | $k_a$ (cm/s)     |                     | 1.1E-08  |          |                     | 4.3E-09  |          |                     | 1.2E-09  |          |
|        | <i>Dif.k</i> (%) | -13.16%             | -5.81%   | 18.97%   | 9.02%               | -23.79%  | 14.78%   | -9.39%              | 10.84%   | -1.45%   |

**Table 5** - Standard deviation and average deviation of the parameter *Dif. k*

| Index              | Sample |       |        |        |        |
|--------------------|--------|-------|--------|--------|--------|
|                    | SN     | SN03  | SN06   | SN09   | SN12   |
| Standard deviation | 4.94%  | 8.14% | 12.51% | 12.88% | 14.32% |
| Average deviation  | 3.80%  | 6.59% | 9.99%  | 10.86% | 11.91% |

**Table 6** -  $k_d/k_{SN}$  values.

| $k_d/k_{SN}$        | Sample |       |       |       |       |
|---------------------|--------|-------|-------|-------|-------|
|                     | SN     | SN03  | SN06  | SN09  | SN12  |
| $\sigma_3 = 20$ kPa | 1.000  | 0.107 | 0.022 | 0.010 | 0.007 |
| $\sigma_3 = 40$ kPa | 1.000  | 0.108 | 0.019 | 0.008 | 0.004 |
| $\sigma_3 = 80$ kPa | 1.000  | 0.087 | 0.014 | 0.004 | 0.002 |

minimum  $k$  for CCL construction (Anderson & Hee, 1995; Mulligan *et al.*, 2001; Allen, 2001). Figure 9 demonstrates that this value was achieved for 3% of bentonite (SN03 sample) under a confining pressure of 40 kPa. Higher bentonite content and  $\sigma_3$  reduced the  $k$  values even more.

The influence of the soil void ratio ( $e$ ) and specific surface ( $SS$ ) on the hydraulic conductivity was evaluated through Figs. 10 and 11. This relation is expressed in the well known Kozeny-Carman Eq. 4, which assumes that  $C$  is a dimensionless factor that takes into account the shape and tortuosity of the soil channels (Carrier, 2003). This equa-

tion incorporates also the fluid parameters  $\gamma$  and  $\mu$  (see Eq. 2).

$$k = \left( \frac{\gamma}{\mu} \right) \left( \frac{1}{C} \right) \left( \frac{e^3}{(1+e)(SS^2)} \right) \quad (4)$$

The plot of void ratio and  $e^3/(1+e)SS^2$  vs.  $k$ , as respectively depicted on Figs. 10 and 11, shows that the best fit equations are non-linear (2<sup>nd</sup> order polynomials). These results corroborate many authors, such as Carman (1937, 1939) and others, who recognize this non-linearity for fine grained materials, as in the present case. On the other hand, Chapuis & Albertin (2003) provide a comprehensive study for the application of the Kozeny-Carman equation, and conclude that this equation may give reasonable estimations for all kind of soils, including silts and clays.

Figure 11 is important not only because it shows the behavior of the hydraulic conductivity for the studied soil, but also because it suggests that the first term in the Kozeny-Carman equation ( $\gamma/\mu C$ ) is not a constant composed by properties of percolating fluid ( $\gamma/\mu$ ) and soil ( $C$ ) constants,

but rather a non-linear term, which depends upon the fluid/soil interaction.

In addition, it is worth to point out that the plot of  $k^{1/2}$  vs.  $1/PI^2$  (Fig. 12) is quite linear, as demonstrated by the high values of the determination coefficients ( $R^2$ ).

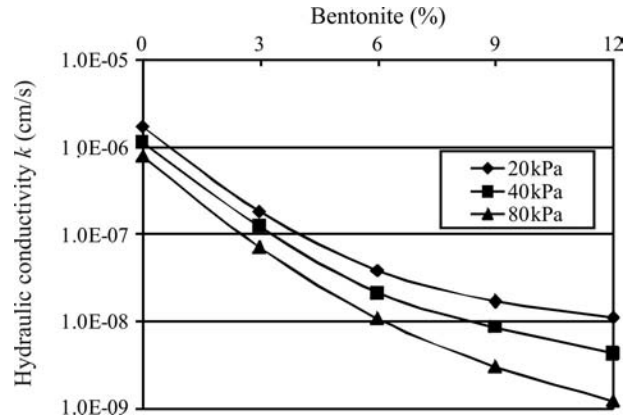


Figure 8 - Hydraulic conductivity ( $k$ ) vs. bentonite content.

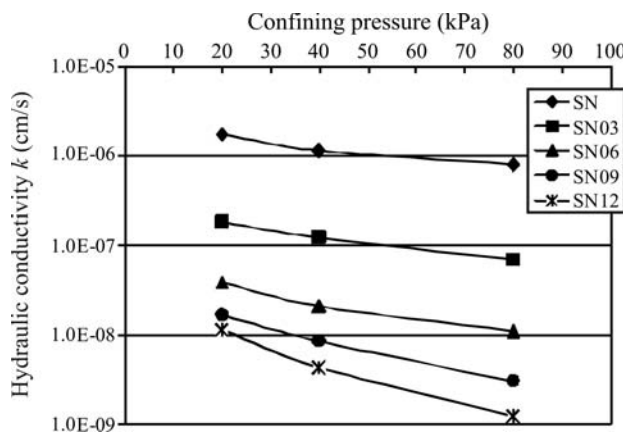


Figure 9 - Hydraulic conductivity ( $k$ ) of the samples under different confining pressures.

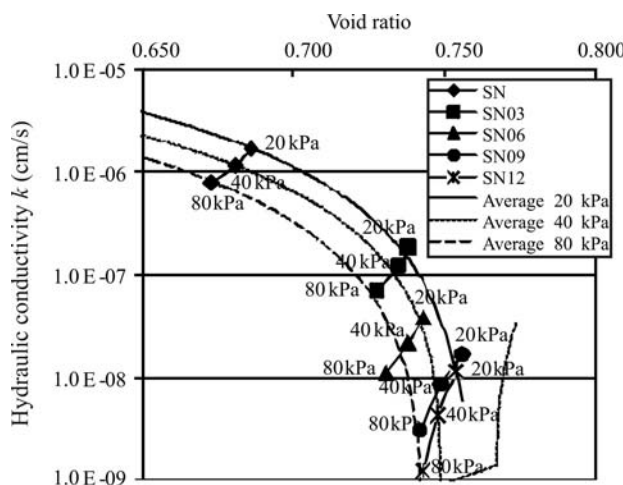


Figure 10 - Hydraulic conductivity ( $k$ ) vs. void ratio ( $e$ ) under different confining pressures.

The result presented in Fig. 12 suggests a fourth order inverse proportionality between the hydraulic conductivity and the plasticity index ( $k \propto 1/PI^4$ ), which justifies the study of the compatibility of the samples under the effect of different solutions, as presented in Chapter 5. As an example, a decrease of 50% in  $PI$  would produce an increase of 16 times in  $k$ .

## 5. Compatibility Tests

### 5.1. Procedures

Compatibility can be defined as the ability of a soil to maintain its original properties after being inundated with different chemical solutions. The long term performance of a CCL strictly depends upon the compatibility (Shackelford, 1994).

Some papers such as Jo *et al.* (2001), Stewart *et al.* (2003), Laird (2006), Katsumi *et al.* (2008), Castellanos *et al.* (2008) and Chalermyanont *et al.* (2009) describe compatibility studies by using laboratory tests to evaluate the influence of different chemical solutions over soil sample. Most of them refer to the *diffusion double layer theory* to explain the soil behavior, as well explicated by Mitchell (1993).

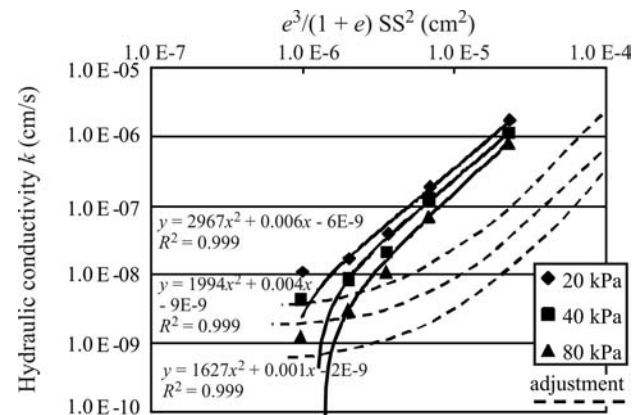


Figure 11 - Hydraulic conductivity ( $k$ ) vs.  $e^3/(1+e) SS^2$ .

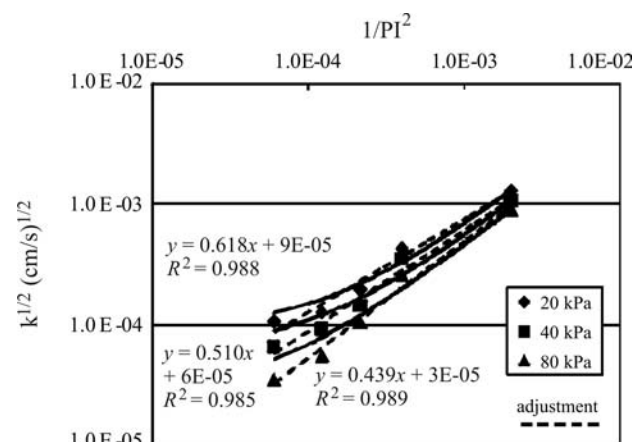


Figure 12 - Root of hydraulic conductivity ( $k^{1/2}$ ) vs.  $(1/PI^2)$ .

In this paper, the compatibility of the samples was assessed in two ways: (1) determination of modified Atterberg Limits for all samples (Shackelford, 1994; Bouazza *et al.*, 2007) and (2) determination of the modified free-swelling index (*FS*) for pure bentonite (BB sample). Free-swelling tests were not performed in mixtures by the fact that these samples do not expand enough to a satisfactory inquiry. The term “modified” refers to the fact that other solutions besides water have been used in the tests. These solutions were: nitric acid ( $\text{HNO}_3$  - pH 3); sodium hydroxide ( $\text{NaOH}$  - pH 11); sodium chloride ( $\text{NaCl}$  - 5 g/L) and ethanol P.A. These solutions may represent some of the most aggressive conditions that a CCL can face in the field.

The modified Atterberg Limits  $\omega_L$  and  $\omega_p$  were determined according to the standards ABNT-NBR6459/84 and 7180/84. The Incompatibility Index (*IC*), as defined in Eq. 5 (Farnezi & Leite, 2007), was used to evaluate the incompatibility (inverse of compatibility) of the samples. According to this equation, the higher the *IC* value, the higher the incompatibility of the soil sample upon the soil solution being tested.

$$IC (\%) = \frac{PI_w - PI_f}{PI_w} \times 100 \quad (5)$$

Being  $PI_w$  the plastic index with water and  $PI_f$  the plastic index with the analyzed fluid.

In turn, the modified free-swelling test (*FS*) consisted of adding gradually (over 30 min) 1 g (dry weight) of bentonite on a graduate test tube filled with 100 mL of distilled water or chemical solution. The *FS* index [mL/g] is determined by direct inquiry in the test tube after 24 h (no agitation) and 48 h (after agitation). This procedure is known worldwide as Foster swelling (Foster 1953, Laird, 2006, Ferreira *et al.*, 2008, Delbem *et al.*, 2010).

## 5.2. Results

Figures 13, 14 and 15 show the Atterberg limits of the SN sample with the increasing bentonite content for the different solutions. An increase in the  $\omega_L$  was noticed for all the solutions as the bentonite content increases (see Fig. 13), while  $\omega_p$  was more stable, exception made for the proportion of 12% of bentonite (Fig. 14). Figure 13 also demonstrates that, relative to water, all the solutions reduced  $\omega_L$  in the following order:  $\text{NaCl} > \text{HNO}_3 > \text{NaOH} > \text{ethanol}$ . As expected, the plastic index curves (Fig. 15) are very similar to those of  $\omega_L$  (Fig. 13). Table 7 presents the results of modified Atterberg limits for the bentonite sample (BB).

Figure 16 curves depict increasing rates of *IC* with the growing proportions of bentonite, which may be interpreted as a negative effect of the bentonite on the compatibility of the samples. This negative effect is less noticeable for the NaCl and ethanol curves. In fact, the SN sample is very sensitive to NaCl solution even without any bentonite

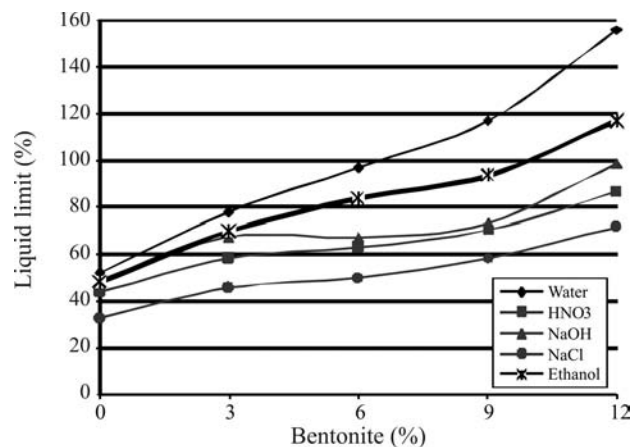


Figure 13 - Liquid limit ( $\omega_L$ ) vs. bentonite content for different chemical solutions.

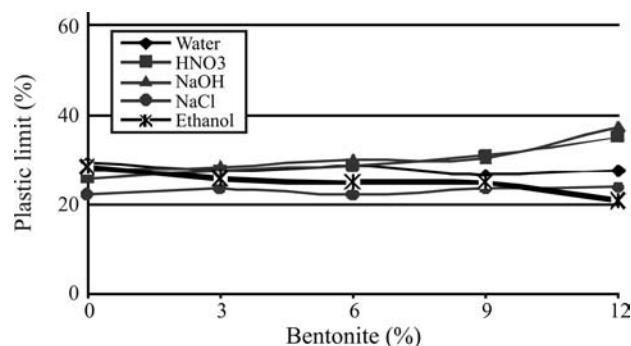


Figure 14 - Plastic limit ( $\omega_p$ ) vs. bentonite content for different chemical solutions.

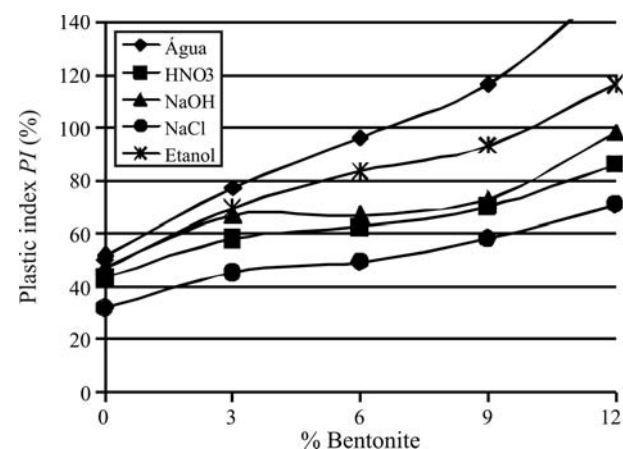


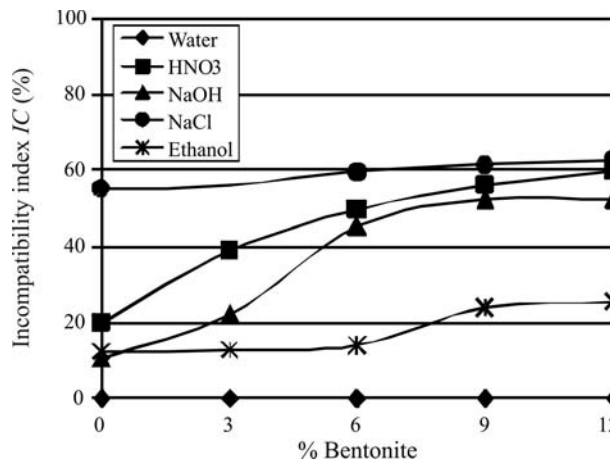
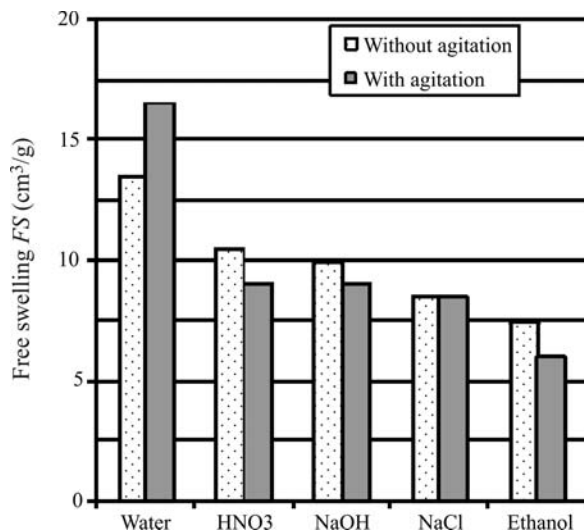
Figure 15 - Plastic index (*PI*) vs. bentonite content for different chemical solutions.

content. Additionally, the NaOH and  $\text{HNO}_3$  curves have similar behavior, approaching the NaCl curve at the highest bentonite contents (9-12%).

The bentonite free swelling (*FS*) results for different solutions are depicted on Fig. 17, including the 24-hour (without agitation) and 48-hour tests (with agitation). All

**Table 7** - Modified Atterberg Limits for bentonite sample.

| Solutions        | $\omega_L$ (%) | $\omega_P$ (%) | $PI$ (%) |
|------------------|----------------|----------------|----------|
| Water            | 682.5          | 90.6           | 591.9    |
| HNO <sub>3</sub> | 495.2          | 140.5          | 354.7    |
| NaOH             | 470.6          | 83.0           | 387.6    |
| NaCl             | 310.9          | 82.8           | 228.1    |
| Ethanol          | 372.9          | 82.6           | 290.3    |


**Figure 16** - Incompatibility index ( $IC$ ) vs. bentonite content for different chemical solutions.

**Figure 17** - Results for free swell tests ( $FS$ ) of BB sample.

the solutions reduced  $FS$  relative to water in the following order:  $H_2O > HNO_3 > NaOH > NaCl > Ethanol$ . The ethanol and NaCl influence on  $FS$  is easily associated to the contraction of the double diffuse layer (DDL) of the clay minerals. The ethanol effect, in turn, is due to its low dielectric constant (24.3) relative to water (80) (see Acar & Olivieri, 1989). For NaCl, the DDL contraction under high salt concentrations is well known (Mitchell, 1993).

Exception made to water and NaCl, the 48-hour tests reduced  $FS$  relative to the 24-hour tests, which may be related to the thixotropic nature of bentonite clay.

## 6. Conclusions

The main conclusions of the flow studies are summarized as follows:

- The addition of bentonite as well as the increase in the confining pressure has led to a significant reduction in hydraulic conductivity. For instance, 12% of bentonite (SN12 sample) reduced  $k$  approximately a thousand times in relation to 0% of bentonite (SN sample) at a confining pressure of 80 kPa. This reduction is potentially increased at higher confining pressures;
- The criteria of  $k = 10^{-7}$  cm/s as the minimum  $k$  for safe barriers was achieved at bentonite contents of 4.5% ( $\sigma_3 = 20$  kPa), 3.5% ( $\sigma_3 = 40$  kPa) and 2.5% ( $\sigma_3 = 80$  kPa);
- The reduction in  $k$  even for higher porosity/void ratios suggests that the adsorbed water did not contributed to the overall flow;

In terms of compatibility, the following conclusions are highlighted:

- Relative to water, all the solutions reduced the plasticity, reflected by the  $\omega_L$  and  $PI$  values, in the following order:  $NaCl > HNO_3 > NaOH > ethanol$ ;
- Except for the NaCl solution, the compatibility of the samples reduces as the bentonite content increases, as demonstrated by the increase in the Incompatibility Index ( $IC$ );
- The free swell tests of the bentonite indicate the following order of impact:  $ethanol > NaCl > NaOH > HNO_3$ .

## Acknowledgments

The authors would like to thank the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) for the grant provided to the first author. Special acknowledgment is also given to the Bentonit União Nordeste S/A, represented by the engineer Pedro Paulo Furtado Gouveia, who kindly donated the bentonite samples used in this research. Finally, the authors appreciate the helpful corrections and suggestions of the Associate Editor and reviewers of this paper.

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## Symbols

- $\mu$ : fluid dynamic viscosity
- $\gamma$ : fluid unit weight

|                                                                        |                                                                                |
|------------------------------------------------------------------------|--------------------------------------------------------------------------------|
| $A$ : soil cross sectional area                                        | $p_2$ : pressure application systems at the over the top of the soil specimens |
| BB: bentonite sample                                                   | $PI$ : plasticity Index                                                        |
| $C$ : dimensionless factor of tortuosity of the soil                   | $PI_f$ : plastic index with the analyzed fluid                                 |
| CEC: cation exchange capacity                                          | $PI_w$ : plastic index with water                                              |
| $Dif.k$ : difference of the $k_i$ value relative to the average $k_a$  | $Q$ : discharge                                                                |
| $e$ : void ratio                                                       | SN03: natural soil sample with 3% of bentonite                                 |
| $e_0$ : initial void ratio of sample                                   | SN06: natural soil sample with 6% of bentonite                                 |
| EC: electrical conductivity of aqueous extract                         | SN09: natural soil sample with 9% of bentonite                                 |
| FS: free-swelling index                                                | SN12: natural soil sample with 12% of bentonite                                |
| GS: specific gravity of solids                                         | SN: natural soil sample                                                        |
| $i$ : hydraulic gradient                                               | SS: specific surface                                                           |
| IC: Incompatibility Index                                              | SS: specific surface                                                           |
| $k$ : hydraulic conductivity                                           | $u$ : pore pressure                                                            |
| $K$ : soil intrinsic permeability                                      | $V_0$ : initial volume of sample                                               |
| $k_a$ : $k$ value of arithmetic average of the three measures          | $w_{ot}$ : optimum moisture content                                            |
| $k_i$ : $k$ value of each soil specimen                                | $\Delta V$ : volume variation of the sample                                    |
| $k_{SN}$ : $k$ value of natural soil sample                            | $\rho_{dmax}$ : maximum dry unit weight                                        |
| $p_1$ : pressure application systems at the base of the soil specimens | $\sigma_3$ : pressure system                                                   |
|                                                                        | $\omega_L$ : liquid limit                                                      |
|                                                                        | $\omega_p$ : plastic limit                                                     |

# Cyclic T-Bar Tests to Evaluate the Remoulded Undrained Shear Strength of the Sarapu  II Soft Clay

G.M.F. Jannuzzi, F.A.B. Danziger, I.S.M. Martins

**Abstract.** The undrained remoulded shear strength of a clay,  $s_{ur}$ , is an important parameter in the design of a number of geotechnical applications. In the case of onshore tests the value of  $s_{ur}$  is generally obtained from vane tests. Recently, cyclic T-bar tests have been used to obtain the  $s_{ur}$  value, especially for offshore applications. Seventeen T-bar cyclic tests in two deployments have been performed at Sarapu  II soft clay test site. In a third deployment only penetration was recorded. The presence of roots has influenced the values of the initial penetration in one of the deployments, as observed in another test site, which is a consequence of the shape of the penetrometer and may be considered a shortcoming of the test. Therefore, to know whether roots have influenced the test results in a site at least two repeatable tests must be performed. If cyclic tests are performed, their results can provide a good indication of the influence of the roots. Considering the vane shear test as reference for obtaining  $s_u$  and  $s_{ur}$ ,  $N_{T-bar}$  obtained from tests not affected by the roots ranged from 8.8 to 10.9, with an average of 9.8, while  $N_{rem, T-bar}$  ranged from 14.1 to 19.5, with an average of 16.3. Therefore  $N_{T-bar}$  values (related to the natural condition) were smaller than  $N_{rem, T-bar}$  (related to the remoulded condition). The equations suggested by Yafate *et al.* (2009) to evaluate the remoulded penetration resistance, the whole degradation curve and the sensitivity, based only on the initial penetration and extraction resistances have provided good results for the Sarapu  II soft clay, except in the case of very shallow depths.

**Keywords:** *in situ* testing, soft clay, T-bar, undrained shear strength, remoulded undrained shear strength, sensitivity.

## 1. Introduction

The remoulded undrained shear strength,  $s_{ur}$ , is an important parameter in the design of suction anchors and for offshore slope stability analyses. For suction anchors, the remoulded undrained shear strength is a key parameter for calculation of the penetration resistance and the under pressure required for installation. The remoulded undrained shear strength also influences the side shear resistance after penetration is completed (*i.e.*, “set-up”) and thereby the holding capacity of an anchor. For offshore slope stability analyses, the remoulded undrained shear strength will influence the failure mechanism and the progressive failure of a potential slide (DeGroot & Lunne, 2007).

In the design of offshore piles the  $s_{ur}$  value (or the sensitivity,  $S_r$ , the ratio between the undisturbed shear strength,  $s_u$  and  $s_{ur}$ ), is used to estimate the friction load during penetration (API, 2004). The  $s_{ur}$  value is also used in the design of torpedo piles (Medeiros Jr., 2010).

In the case of onshore tests the value of  $s_{ur}$  is generally obtained from vane tests. Recently, cyclic T-bar tests have been used to obtain the  $s_{ur}$  value, especially for offshore applications.

This paper analyses cyclic T-bar tests performed at Sarapu  II soft clay test site. Comparisons are made with values obtained from electrical vane tests.

## 2. T-bar tests

### 2.1. Historical

T-bar tests have been originally developed to be used in centrifuge testing at the University of Western Australia (UWA) by Stewart & Randolph (1991), aiming at the determination of a continuous profile of the undrained shear strength of soft clays. The test consisted of the penetration of a cylindrical horizontal bar, as shown in Fig. 1, at a rate of 3 mm/s.

This new test would combine the advantages of the CPT or CPTU (which gives a continuous profile of “strength”), and the vane test (which gives an “exact” or direct measure of shear strength) (Stewart & Randolph, 1991).

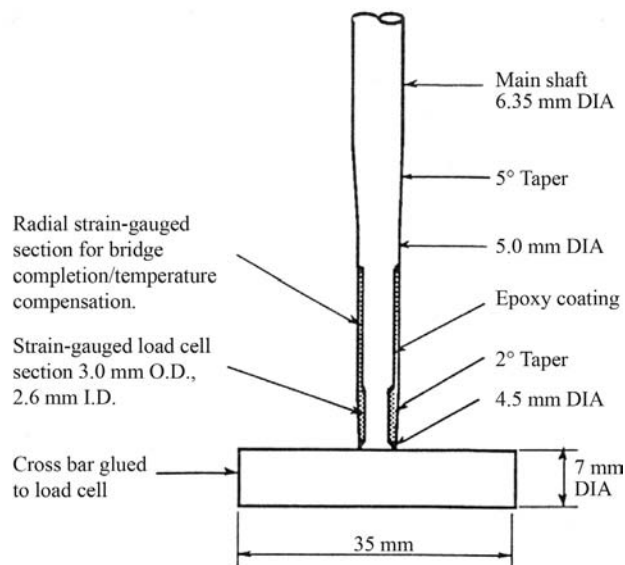
The T-bar was firstly used in the field in 1994 (Stewart & Randolph, 1994), in Burswood, Australia, and comprised a 50 mm in diameter and 200 mm long aluminium bar. The same rate of penetration used in the piezocone test, 20 mm/s, was also used in the T-bar test. Later the T-bar was used offshore, also in Australia, and changed its dimensions to a 40 mm in diameter and 250 mm long bar (Randolph *et al.*, 1998). These dimensions are included in the only standard that covers T-bar penetration testing, the NORSOK G-001 (Standards Norway, 2004) (Lunne *et al.*, 2011).

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Submitted on October 31, 2011; Final Acceptance on November 21 2012; Discussion open until April 30, 2013.



**Figure 1** - Schematic diagram of the T-bar (Stewart & Randolph, 1991).

The resistance during penetration,  $q_m$ , is obtained from the load measured at the load cell divided by the projected area of the T-bar, *i.e.* 100 cm<sup>2</sup> in the case of *in situ* tests.

The main advantage of the T-bar test with respect to the CPTU test was thought to be that the interpretation of the T-bar test is based on the analytical solution of Randolph & Houlsby (1984), which shows that the penetration resistance does not depend on the rigidity index  $I_r (= G/s_u$ , where  $G$  is the shear modulus), as in the case of the piezocone (*e.g.*, Levadoux, 1980, Teh, 1987, Teh & Houlsby, 1991).

Other advantages of the T-bar are (i) improved accuracy in soft soils due to a larger penetrometer projected area and (ii) minimal correction for overburden stress (*e.g.*, Yafrate *et al.*, 2009).

Cyclic tests have been performed by Hefer & Neubecker (1999), aiming at the evaluation of the undrained remoulded shear strength. Cycles of penetration and extraction over a fixed 0.5 m depth interval were performed until it was apparent that a true residual (as mentioned by Hefer & Neubecker, 1999) or remoulded soil strength had been achieved.

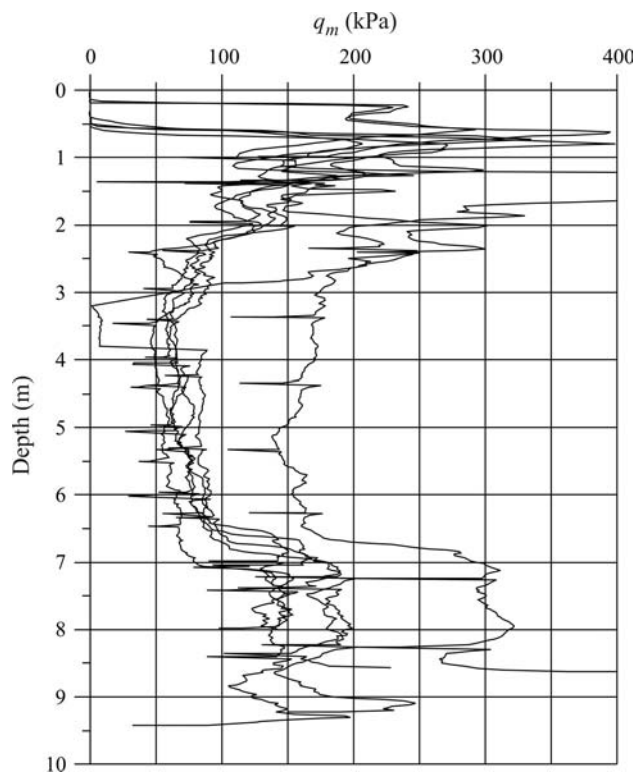
T-bar cyclic tests were initially performed during extraction. Then this procedure was changed, and the cyclic tests were recommended to be performed during penetration phase, because partial consolidation of the soil around the push rod would result in higher extraction and remoulded resistances being measured if the cyclic penetration test is carried out during the extraction phase of the test (*e.g.*, Lunne *et al.*, 2011). According to those authors it is recommended that ten cycles of penetration and extraction through a minimum stroke of 0.15 m should be undertaken. Yafrate *et al.* (2009) mentioned that full strength degrada-

tion occurs within five to ten cycles. The penetration and extraction rate for the cyclic test should be the same as for the penetration phase, *i.e.* 20 mm/s should be used.

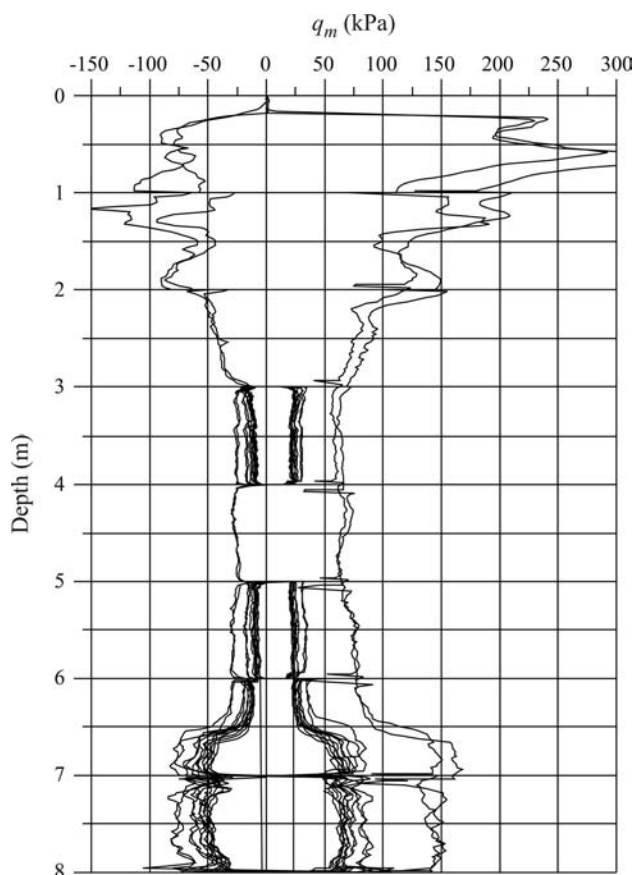
To the authors' knowledge, the first T-bar tests in Brazil were carried out in a soft clay deposit at the site where the 2007 Pan American Games Athletes' Village was to be built, at Barra da Tijuca, Rio de Janeiro (Macedo, 2004, Almeida *et al.*, 2006, Danziger, 2007). Two series of tests were performed with a T-bar penetrometer built from a COPPE piezocone penetrometer. Penetration and extraction resistance have been measured in the first series, where four tests were performed. Cyclic tests - during extraction phase - were conducted in the second series, where two tests have been carried out. Five cycles of penetration have been performed through a 1 m stroke, in the intervals 3-4 m, 5-6 m, 6-7 m, 7-8 m. The tests were part of a joint research project between NGI (the Norwegian Geotechnical Institute) and COPPE/Federal University of Rio de Janeiro. The penetration resistance of the first series of tests is shown in Fig. 2. Cyclic tests are shown in Fig. 3. Penetration values are plotted as positive values while negative values are used in the case of extraction.

## 2.2. Interpretation

As mentioned before, the interpretation of the T-bar test was based on the analytical solution of Randolph & Houlsby (1984). Classical plasticity theory was used to de-



**Figure 2** - Penetration resistance from T-bar tests at Pan American Games Athletes' Village test site (adapted from Macedo, 2004).



**Figure 3** - Cyclic tests at Pan American Games Athletes' Village test site (adapted from Macedo, 2004).

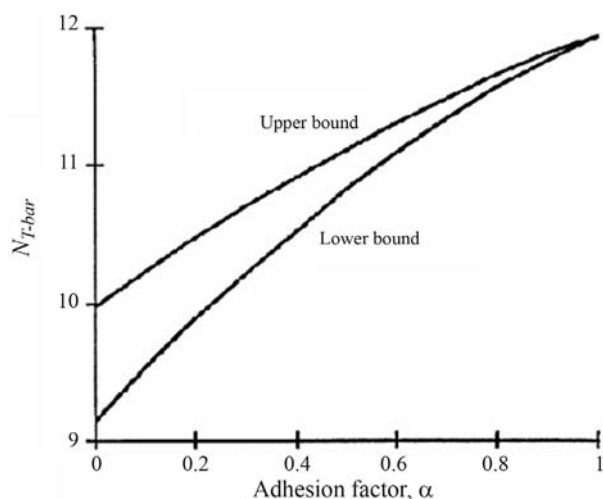
rive exact solutions for the limiting lateral resistance of a circular pile (with infinite length) embedded in a saturated clay. Lower bound and upper bound approaches were used, and the final failure load per unit length of pile,  $P$ , normalized by the pile diameter,  $d$ , and  $s_u$  was obtained. The corresponding ratio is the  $N_{T-bar}$  factor, Eq. 1. The solution obtained by Randolph & Houlsby (1984), amended by Murff *et al.* (1989), has been presented by Stewart & Randolph (1991), as shown in Fig. 4.

$$\frac{P}{s_u d} = N_{T-bar} \quad (1)$$

The resistance during penetration,  $q_m$ , is obtained from Eq. 2

$$q_m = \frac{P}{d} \quad (2)$$

The analytical factor  $N_{T-bar}$  depends on the surface roughness of the cylinder, described by its adhesion factor,  $\alpha$ . According to Randolph & Houlsby (1984) and Stewart & Randolph (1991), the adhesion factor very difficultly would reach 0 (perfectly smooth bar) or 1 (perfectly rough), thus a value of 10.5 was suggested for  $N_{T-bar}$ . According to



**Figure 4** - Variation of  $N_{T-bar}$  with surface roughness (Stewart & Randolph, 1991).

Stewart & Randolph (1991), the use of  $N_{T-bar} = 10.5$ , associated to the very narrow range of possible factors for  $N_{T-bar}$  (9.14 to 11.94), implies in a maximum error of 13%.

The initial papers about the T-bar mentioned that the *in situ* vertical stress is equilibrated across the T-bar, and thus there is no requirement to include a correction for the ambient stress level, unlike the CPT (Stewart & Randolph, 1994). Thus, the  $N_{T-bar}$  factor was defined in terms of the total value of the T-bar penetration, or  $q_m$  (Eq. 2), as shown above. Watson *et al.* (1998) did mention a net value of penetration resistance, but also that there is no need to correct for overburden stress, and the measured penetration resistance is equal to the net penetration resistance.

Later, Chung & Randolph (2004) and Randolph (2004) suggested the correction shown in Eq. 3 to obtain  $q_{net}$ .

$$q_{net} = q_m - [\sigma_{vo} - u_o (1 - a_r)] \frac{A_s}{A_p} \quad (3)$$

where  $\sigma_{vo}$  = total vertical stress;  $u_o$  = hydrostatic pore pressure;  $a_r$  = load cell area ratio;  $A_p$  = the projected cross-sectional area of the T-bar and  $A_s$  = the cross-sectional area of the connection shaft (or push rods). Equation 3 must be used to correct both penetration and extraction resistances, inclusively during cyclic tests (Lunne *et al.*, 2011).

It follows that the  $N_{T-bar}$  factor must be defined as in Eq. 4

$$N_{T-bar} = \frac{q_{net}}{s_u} \quad (4)$$

It must be pointed out that the value of  $q_{net}$  for the T-bar test is similar to the  $q_{net}$  obtained from the piezocone test, Eq. 5

$$q_{net} = q_t - \sigma_{vo} \quad (5)$$

where  $q_t$  is the cone resistance corrected for unequal end area, Eq. 6, from Campanella *et al.* (1982).

$$q_t = q_c + (1-a)u_2 \quad (6)$$

where  $q_c$  is the measured cone resistance,  $a$  the cone area ratio and  $u_2$  the pore pressure measured at the cone shoulder.

Chung & Randolph (2004) verified that the correction for the T-bar is far less significant than that for the piezocone. This was largely due to the very small  $A_s/A_p$  ratio, typically 0.1 to 0.2 (Randolph, 2004).

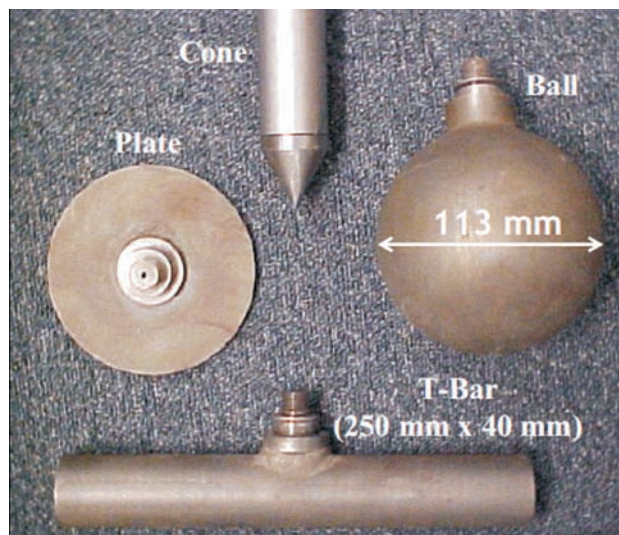
Yafrate *et al.* (2009) mentioned that  $u_0$  in Eq. 3 should be replaced by the value of  $u_2$  when it is available, which may occur when the T-bar is just the replacement of the cone tip from a piezocone penetrometer.

It must be pointed out that the theoretical analysis previously shown was based on plasticity solutions for simple rate independent, perfectly plastic soil models with isotropic strength (Randolph & Andersen, 2006). Recent numerical analysis performed by those authors, where anisotropy, rate dependency of shear strength and strain softening have been considered, have shown that all these factors do affect  $N_{T-bar}$ .

Data from ten sites, both onshore and offshore have been used by Low *et al.* (2010, 2011) to evaluate the effect of soil characteristics on piezocone, T-bar and ball penetration tests, as well as the corresponding  $N$  factors. *In situ* vane tests and laboratory tests (triaxial compression, triaxial extension and direct simple shear tests, providing respectively  $s_{uc}$ ,  $s_{ue}$ ,  $s_{uSS}$ ) have been used as references for the analysis. It was overall found (Low *et al.*, 2010) that the only significant trend to emerge from the database is the effect of the rigidity index on the cone factor  $N_{KT}$ , which increases with increasing  $I_r$ , as theoretically predicted (*e.g.*, Teh & Houlsby, 1991). Low *et al.* (2010) mentioned that, at least for the soils with  $S_r$  less than 6 that dominate the database, T-bar and ball penetration tests may potentially prove more reliable than CPTU in estimating  $s_{uave}$  (the average of  $s_{uc}$ ,  $s_{ue}$  and  $s_{uSS}$ ) or  $s_{uavane}$  but the reverse is probably true for estimation of  $s_{uc}$ . Low *et al.* (2011) concluded that although theoretical solutions for penetrometers in isotropic, rate-independent and non-softening soils generally predict the trends of the field data, there are still discrepancies between the theoretical predictions and measured values. Further study is required to improve the theoretical solutions.

### 2.3. Other full-flow penetrometers

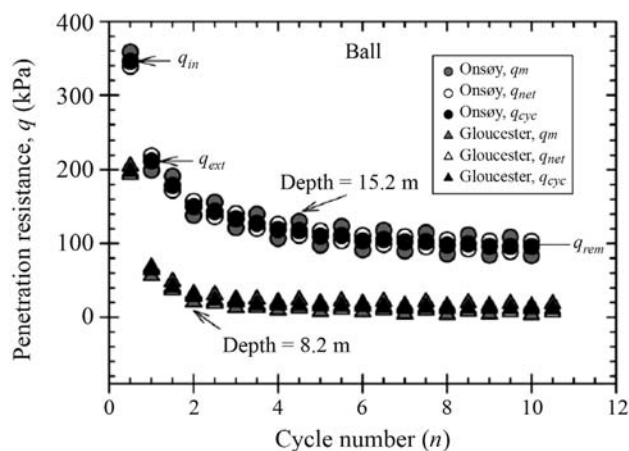
The T-bar is named a “full-flow” penetrometer because soil flows around the penetrometer during the penetration process, with soil occupying much of the same volume it did initially. This contrasts the cone penetrometer, where all soil is permanently displaced (Yafrate *et al.*, 2009). Other full-flow penetrometers are the ball penetrometer and the plate penetrometer. Figure 5 shows a schematic view of the “full-flow” penetrometers together with a regular 10 cm<sup>2</sup> piezocone.



**Figure 5** - T-bar, ball, plate and 10 cm<sup>2</sup> cone penetrometers (Randolph, 2004).

### 2.4. Degradation during cyclic testing

Cyclic tests have been presented in Fig. 3, where penetration and extraction resistances were plotted vs. depth. Another way of representing cyclic test results is to average the central part of each cycle stroke - to avoid the influence of conditions at the extreme of the cyclic zone - and plot both penetration and the modulus of the extraction values vs. number of cycle, which consists the so-called degradation curve (*e.g.*, Lunne *et al.*, 2011). It has been initially suggested that the cycle number for the initial penetration should be taken as 0.25 and initial extraction taken as 0.75 and so forth (Randolph *et al.*, 2007, Lunne *et al.*, 2011). Yafrate *et al.* (2009) mentioned that conventional practice is to present the initial penetration as cycle 0.5 and initial extraction as cycle 1. The degradation curves for Onsøy and Gloucester clays and the ball penetrometer using this representation are shown in Fig. 6. Both measured ( $q_m$ ) and cor-



**Figure 6** - Cyclic ball penetrometer degradation curves from Onsøy and Gloucester test sites (DeJong *et al.*, 2010a).

rected ( $q_{net}$ ) values are shown in the figure. The values corresponding to the initial penetration are named  $q_{in}$  and those corresponding to the initial extraction  $q_{ext}$ .

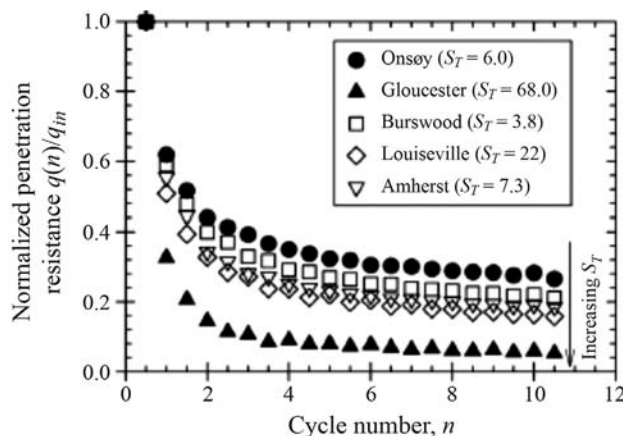
As pointed out by Yafrate *et al.* (2009), during cyclic testing the magnitude of penetration and extraction resistance (both  $q_m$  and  $q_{net}$ ) is not generally equal under remoulded conditions. To create a smooth degradation curve one-half of the difference between penetration and extraction resistance in the remoulded condition (termed the cyclic offset) is added to (or subtracted from) each value of net penetration resistance, resulting in  $q_{cyc}$  values in Fig. 6. According to those authors, the reason for this offset is not yet clear, and a number of factors can contribute to it.

To compare test results from different depths and locations, Yafrate *et al.* (2009) have normalized the penetration resistance values in the cycles,  $q(n)$ , with respect to the initial values,  $q_{in}$ . Test results from different test sites are presented in Fig. 7, which shows that the shape of the curve is affected by the soil sensitivity, the higher the sensitivity the faster the resistance degradation.

The normalized cyclic degradation curve inherently contains information regarding the soil sensitivity and the rate at which the soil strength reduces (strain softening), Yafrate *et al.* (2009). The soil sensitivity is related to the ratio  $q_{rem}/q_{in}$ , while the rate of softening is related to the ratio  $q_{ext}/q_{in}$ . It can be seen from Fig. 7 that the penetration resistance degrades more rapidly at the Gloucester test site (3 cycles to  $q_{rem}$ ) than at the Onsøy test site (8 to 10 cycles to  $q_{rem}$ ) due to the higher sensitivity at Gloucester. The rate of strain softening and sensitivity are interrelated.

Yafrate *et al.* (2009) suggested Eq. 7 to estimate the remoulded shear strength based only on initial penetration  $q_{in}$  and extraction  $q_{ext}$  values, *i.e.*, without the need of performing the cyclic test, aiming at initial estimates.

$$\frac{q_{rem}}{q_{in}} = \left( \frac{q_{ext}}{q_{in}} \right)^{2.8} \quad (7)$$



**Figure 7** - Normalized cyclic degradation curves for various sites (Yafrate *et al.*, 2009).

The whole degradation curve can also be estimated based only on  $q_{in}$  and  $q_{ext}$  values, according to Eq. 8 below, valid for  $n \geq 1$  (Yafrate *et al.*, 2009).

$$\frac{q(n)}{q_{in}} = \left( \frac{q_{ext}}{q_{in}} \right)^{2.8} + \left( \frac{q_{ext}}{q_{in}} - \left( \frac{q_{ext}}{q_{in}} \right)^{2.8} \right) e^{-3(n-1)/(9.6(q_{ext}/q_{in}))} \quad (8)$$

## 2.5. Estimation of soil sensitivity

The soil sensitivity is not equal to the ratio  $q_{in}/q_{rem}$ , as one would expect to be, but rather can be estimated according to Eq. 9 (Yafrate *et al.*, 2009). Equation 9 was based on experimental values (Fig. 8), where the reference sensitivity was obtained from field vane tests. Equation 9 indicates that even with a significant number of cycles the T-bar is not able to completely remould the soil in the same way as the vane test does. Actually, *e.g.*, Randolph & Andersen (2006) and Lunne & Andersen (2007) have shown that different methods provide different values of the remoulded shear strength. Rate dependency of the remoulded shear strength in a similar way as intact shear strength may at least partly explain the mentioned differences (Lunne & Andersen, 2007).

$$S_T = \left( \frac{q_{in}}{q_{rem}} \right)^{1.4} \quad (9)$$

Due to the interrelationship between the rate of initial strain softening and the soil sensitivity, the soil sensitivity can also be estimated from the  $q_{in}/q_{ext}$  ratio, *i.e.* without the need of performing the cyclic test, according to Eq. 10, also based on field vane tests to obtain the sensitivity values.

$$S_T = \left( \frac{q_{in}}{q_{ext}} \right)^{3.7} \quad (10)$$

## 2.6. Estimation of remoulded bar factor

The  $N_{T-bar}$  factor for the remoulded condition,  $N_{rem, T-bar}$  is defined as

$$N_{rem, T-bar} = \frac{q_{rem}}{s_{ur}} \quad (11)$$

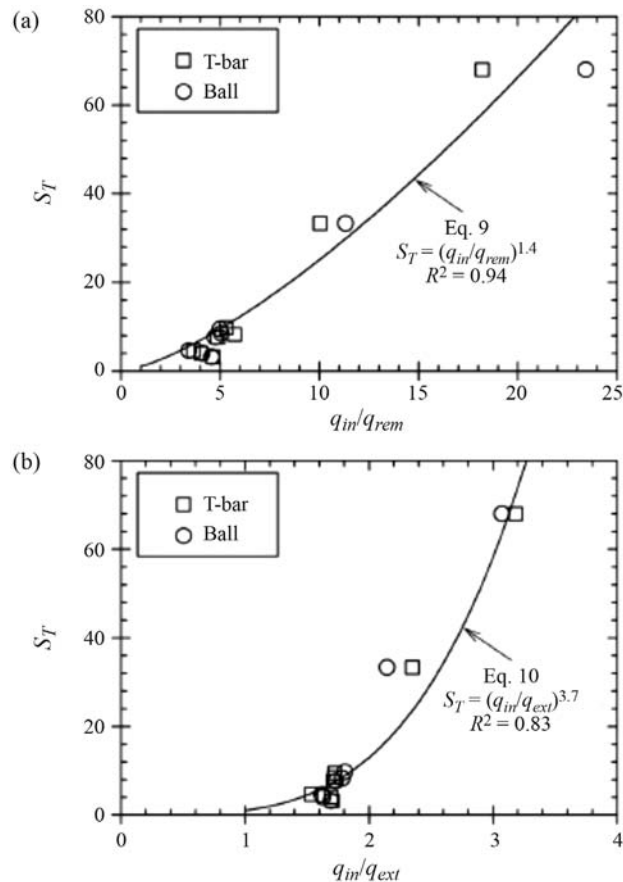
Yafrate *et al.* (2009) verified a trend of  $N_{rem, T-bar}$  to increase with the increase of sensitivity, according to Eq. 12.

$$N_{rem, T-bar} = 12 + \frac{5.5}{1 + \left( \frac{S_T}{6} \right)^{-3}} \quad (12)$$

As a consequence of considering the reference shear strength both in undisturbed and remoulded condition from the vane test, and Eq. 9, it follows that  $N_{T-bar}$  and  $N_{rem, T-bar}$  are not equal.

## 3. The Test Site

The Sarapu soft clay test site has been used since the 70's as a research site, and a number of *in situ* and laboratory tests have already been performed (*e.g.*, Lacerda *et al.*,

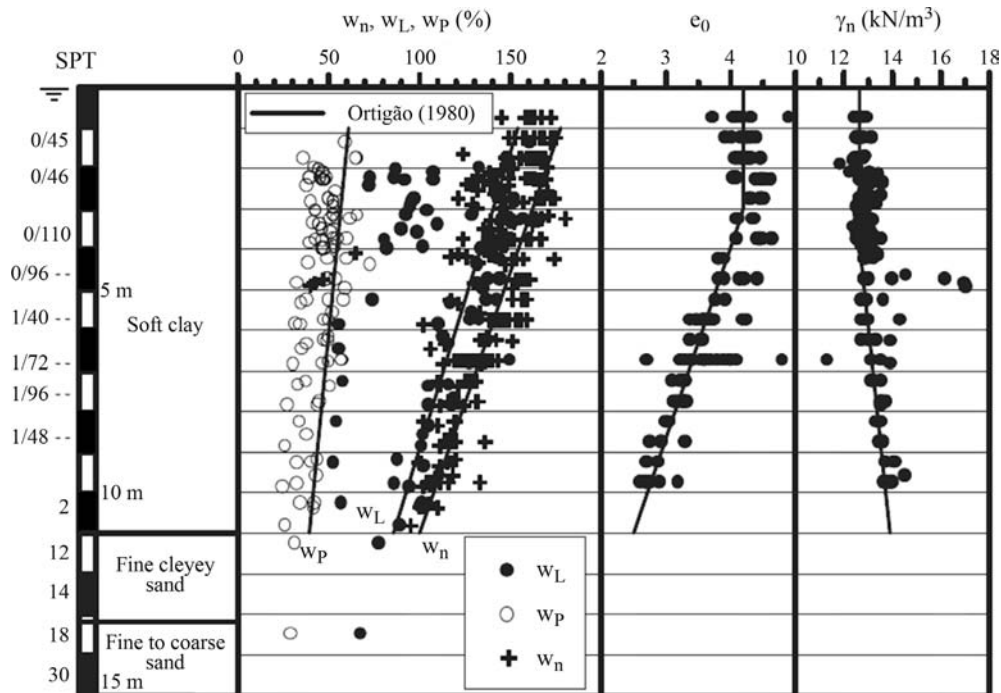


**Figure 8** - Sensitivity as a function of (a)  $q_{in}/q_{rem}$  and (b)  $q_{in}/q_{ext}$  (Yafrate *et al.*, 2009).

1977, Werneck *et al.*, 1977, Ortigão *et al.*, 1983). A comprehensive report about the deposit has been provided by Almeida & Marques (2002). Geotechnical characteristics of the soil are included in Figs. 9 and 10, based on investigations carried out near the trial embankments sites. The very soft organic clay layer is about 11 m thick, and overlies sand layers. The plasticity index (IP) of the Sarapu clay decreases with depth, from around 100% to 50%. Stress history and compressibility characteristics of the deposit are shown in Fig. 10.

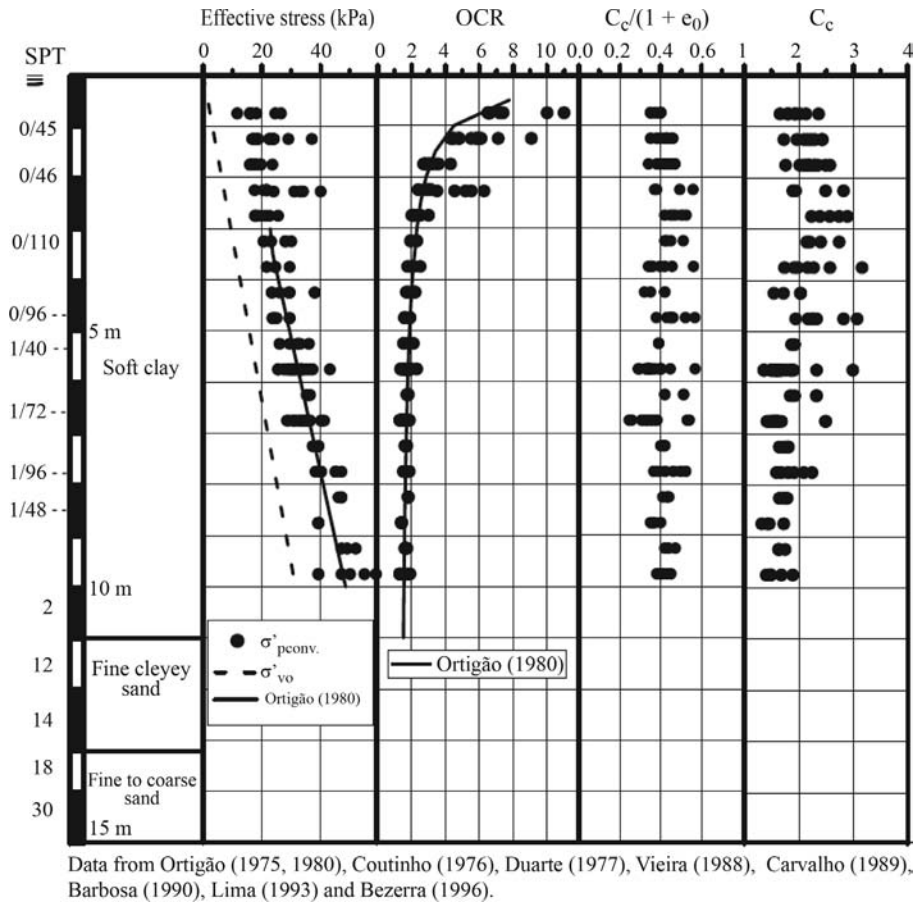
In the last fifteen years, however, security reasons have prevented the use of the test site. A new area (named Sarapu II) in the same deposit, 1.5 km from the previous area and inside of a Navy Facility, has been used since then (Fig. 11). Two researches on pile behaviour have been carried out at Sarapu II site (Alves, 2004, Francisco, 2004, Alves *et al.*, 2009). The initial tests with the torpedo-piezcone (Porto *et al.*, 2010, Jannuzzi *et al.*, 2010, Henriques Jr. *et al.*, 2010) have already been performed at Sarapu II test site.

Although the whole deposit can be considered fairly homogeneous in horizontal directions, a number of *in situ* tests have been performed in this new area. In fact, 6 deployments of SPT's (performed at each meter in Brazil), 7 CPTU's, 51 vane tests (in 5 deployments) and 4 T-bar tests have been performed (Jannuzzi, 2009). The very soft clay layer in this particular area varies from 6.5 m to 10 m. This new area is been used by the Research Center of the Brazilian Oil Company (CENPES/PETROBRAS) and Federal



Data from Ortigão (1975, 1980), Coutinho (1976), Duarte (1977), Collet (1978), Vieira (1988), Barbosa (1990) and Lima (1993).

**Figure 9** - Characteristics of Sarapu soft clay deposit (Almeida & Marques, 2002).



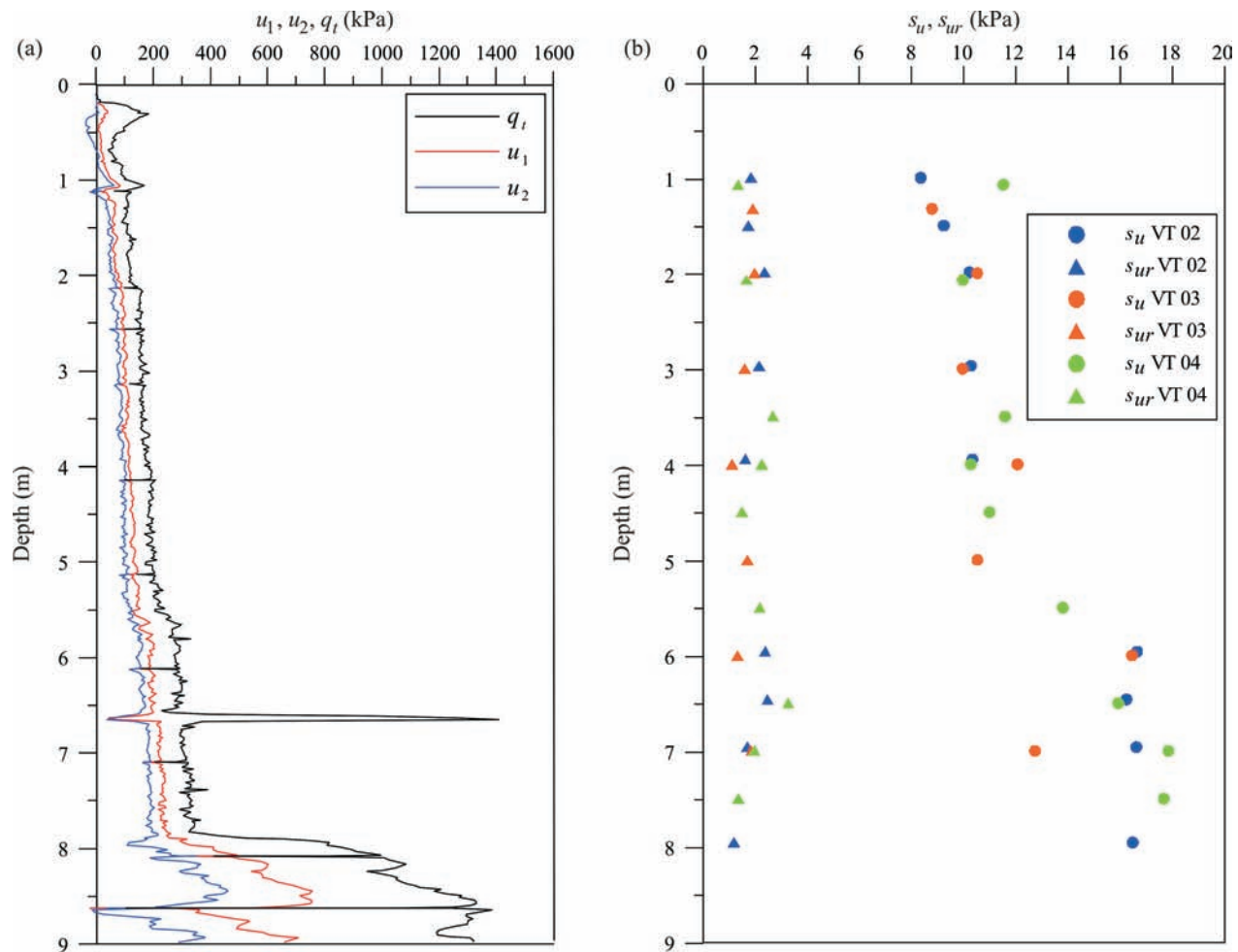
**Figure 10** - Stresses and compressibility parameter profiles (Almeida & Marques, 2002).



**Figure 11** - Sarapuí II test site with respect to the early Sarapuí I test site.

University of Rio de Janeiro as a state-of-the-art test site on very soft organic clay. Laboratory tests (triaxial and direct simple shear) on very high quality samples will be performed in 2013. Instrumented model torpedo-anchors will

be tested in the same area. Fig. 12a shows corrected cone resistance  $q_c$ , pore-pressures at the cone shoulder  $u_2$  and cone face  $u_1$  vs. depth from a typical piezocone test. It can be seen that the very soft clay layer is around 8 m deep, and



**Figure 12** - (a) Cone resistance and pore pressure from typical piezocone test; (b) undrained shear strength and remoulded undrained shear strength from vane tests in 3 deployments; Sarapuí II soft clay test site (Jannuzzi, 2009).

a clayey-silt layer underlies the very soft clay. Fig. 12b shows  $s_u$  and  $s_{ur}$  values obtained from 3 vane test deployments. The equipment used is able to measure the torque close to the blade, aiming at minimizing the rod friction, which was developed in a joint research project among the Federal University of Rio de Janeiro, the Federal University of Pernambuco and Grom Eng. (e.g., Nascimento, 1998, Oliveira, 2000, Coutinho *et al.*, 2000, Crespo Neto, 2004). Further details of the *in situ* tests can be obtained in Jannuzzi (2009).

#### 4. Tests Performed

Four T-bar tests have been performed, three of them in the natural soil and one under an existing embankment, in order to verify the ability of the T-bar to identify the influence of the embankment on the soft material (Jannuzzi *et al.*, 2012). Values of the measured penetration resistance,  $q_m$ , for the three tests performed in the natural soil are presented in Fig. 13. It can be seen that the values of  $q_m$  corresponding to T-bar 3 are greater than the other tests, which was attributed to the shape of the T-bar allowing the exist-

ing roots of the vegetation to be pushed together with the penetrometer, increasing the corresponding resistance (see Fig. 14). It must be pointed out that the roots layer is roughly 30 cm thick, where the upper half is composed by thick roots (few millimeters, reaching in some cases 3 cm in diameter) and the lower half typically 1 mm in diameter. Similar phenomenon was verified by Macedo (2004), see also Almeida *et al.* (2006) and Danziger (2007). This means that when performing T-bar tests in places where there is an intense presence of roots, at least two tests with good repeatability must be performed in order to be sure that the results are not affected by the roots. Another way of checking whether roots are influencing the penetration resistance is through cyclic test results, as shown later.

Seventeen cyclic tests were conducted during extraction phase in T-bar 2 and 3, which are plotted vs. depth in Figs. 15 and 16, and summarized in Table 1. Each cyclic test consisted in 5 or 6 cycles. Due to localized malfunctioning of the device that holds the rods during penetration and extraction - allowing them to have some sliding, thus preventing an accurate control of the depth -, test T-bar 2-7

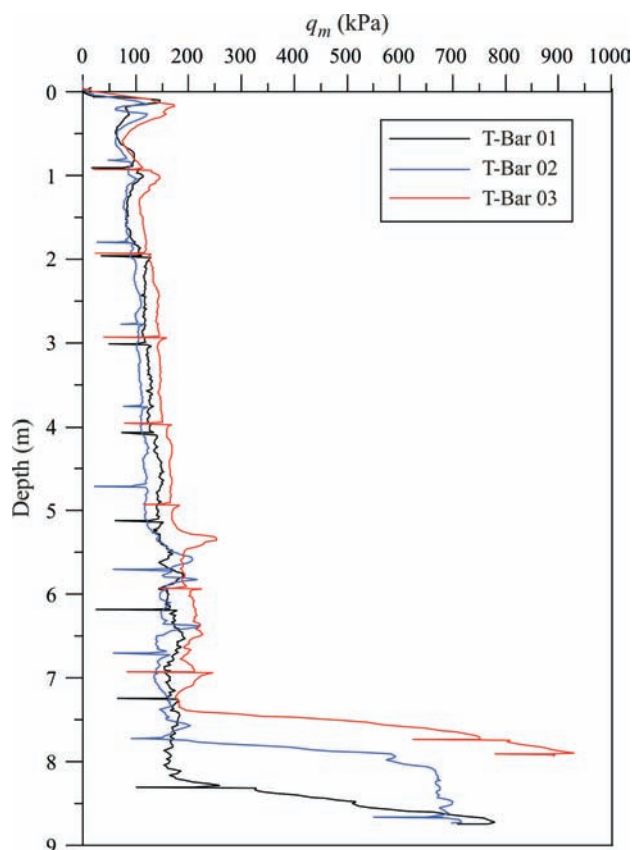


Figure 13 - Measured penetration resistance,  $q_m$ , vs. depth.

Table 1 - Cyclic tests performed.

| Test      | Nominal interval cycled (m) | Average depth (m) | Number of cycles |
|-----------|-----------------------------|-------------------|------------------|
| T-bar 2-1 | 0.00-0.73                   | 0.36              | 6                |
| T-bar 2-2 | 0.73-1.73                   | 1.23              | 6                |
| T-bar 2-3 | 1.73-2.73                   | 2.23              | 6                |
| T-bar 2-4 | 2.73-3.73                   | 3.23              | 6                |
| T-bar 2-5 | 3.73-4.73                   | 4.23              | 6                |
| T-bar 2-6 | 4.73-5.73                   | 5.23              | 6                |
| T-bar 2-7 | 5.73-6.73                   | 6.23              | 6                |
| T-bar 2-8 | 6.73-7.73                   | 7.23              | 5                |
| T-bar 2-9 | 7.73-8.73                   | 8.23              | 5                |
| T-bar 3-1 | 0.00-0.92                   | 0.46              | 6                |
| T-bar 3-2 | 0.92-1.92                   | 1.42              | 6                |
| T-bar 3-3 | 1.92-2.92                   | 2.42              | 6                |
| T-bar 3-4 | 2.92-3.92                   | 3.42              | 6                |
| T-bar 3-5 | 3.92-4.92                   | 4.42              | 6                |
| T-bar 3-6 | 4.92-5.92                   | 5.42              | 6                |
| T-bar 3-7 | 5.92-6.92                   | 6.42              | 6                |
| T-bar 3-8 | 6.92-7.92                   | 7.42              | 6                |



Figure 14 - T-bar in the beginning of a test at Sarapuí II test site, where the intense presence of roots can be noted.

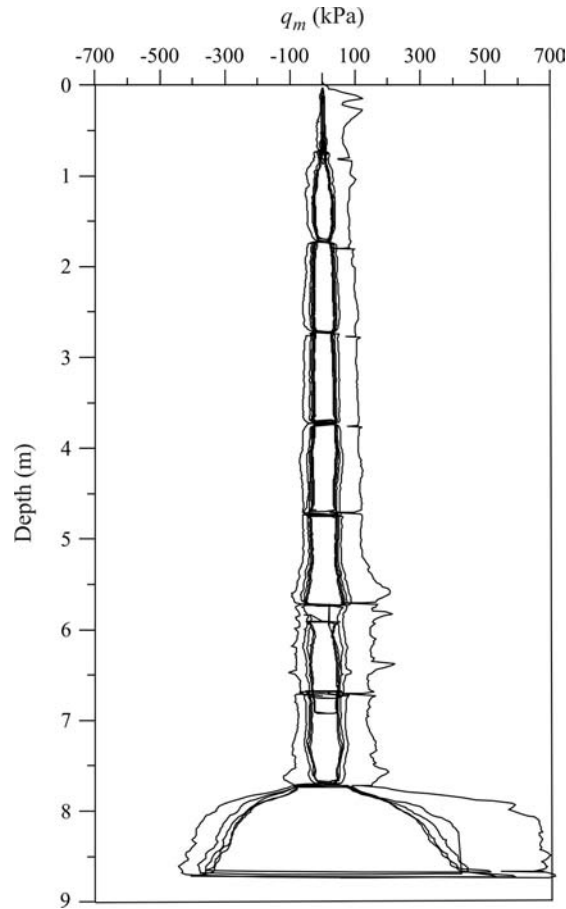
was not cycled in the same interval, *i.e.* did not have the same initial and final depths in each cycle. Besides, the procedure used to end up each test was not accurate enough to guarantee the exact same depth, thus few centimeters in difference may be found in the initial and final depths from one cycle to another. This problem has not, however, produced errors in the test results, since the 10 cm in the middle of the penetration interval of 1 m were considered to obtain the average value. It must be emphasized that the lack of accuracy in the beginning-end of each cyclic test is a regular occurrence, *i.e.* it is not a particular occurrence of the tests performed at Sarapuí II.

It can be observed from Figs. 15 and 16 that the deepest test was performed entirely in the clayey-silt layer in the case of T-bar 2, while the corresponding test in the case of T-bar 3 involved both the very soft clay and the clayey-silt material. The magnification of two cyclic tests is presented in Figs. 17 and 18.

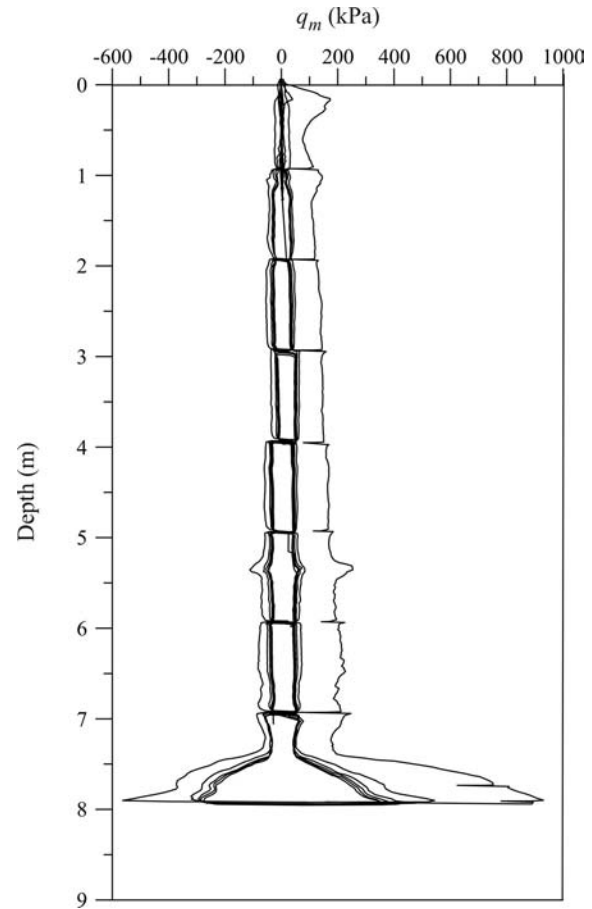
## 5. Analysis and Discussion

The values of  $q_m$  and  $q_{net}$  in two degradation curves are shown in Figs. 19 and 20, where the initial penetration was assigned cycle number 0.5, initial extraction cycle number 1, second penetration cycle number 1.5 and so forth. In both cases, the trend of  $q_m$  being greater in the penetration than in extraction was found. This behaviour was found in all tests performed. As far as  $q_{net}$  is concerned, in most cases the same trend found for  $q_m$  was found, *i.e.*,  $q_{net}$  was greater in the penetration than in extraction. However, few cases have shown a different trend, with  $q_{net}$  being smaller in the penetration than in extraction, which is shown in Fig. 20. The reason for this behaviour still deserves investigation.

The normalized cyclic degradation curves ( $q_{cyc}$  vs. cycle number) of all tests are presented in Figs. 21 and 22, respectively for T-bar 2 and T-bar 3. It is interesting to note that the degradation is much faster and greater in the tests performed at the smallest depths (T-bar 2-1 and T-bar 3-1),



**Figure 15** - T-bar 2 - cyclic tests.



**Figure 16** - T-bar 3 - cyclic tests.

while the tests performed at the greatest depths (T-bar 2-9 and T-bar 3-8) presented the smallest degradation. All other tests have presented a similar normalized behaviour.

The behaviour associated with the tests performed at the smallest depths may represent the real soil behaviour or not. The first case would correspond to soil sensitivity higher at those depths than at the other depths. To evaluate this hypothesis vane tests would have been performed. However, vane tests are not available at such small depths. Two explanations may be provided for the second case. The first one is related to the presence of the roots, *i.e.* the value of  $q_{in}$  (initial penetration value) is mostly due to the resistance offered by the roots, which is removed when the cyclic test is performed. Another possible explanation is related to the very low effective stresses at those test depths. In this case the full-flow mechanism may not occur (DeJong *et al.*, 2010). More research is needed to properly investigate this subject. This is indeed an issue, since T-bar tests are very often used as an investigation tool in the design of pipelines in very soft soils, where the remoulded shear strength is an important parameter.

As far as the deepest tests are concerned, they have indicated that this clayey-silt layer has a smaller sensitivity than the soft clay material. This hypothesis cannot be

checked, since vane tests are not available due to the difficulties in penetrating the vane blade in the silty material. Another possible explanation is also related to the non occurrence of the full-flow mechanism, which might happen in stiff soils, where an open cavity or “slotting” may occur (DeJong *et al.*, 2010).

Although a similar trend was found in all cyclic tests for T-bar 2 and T-bar 3 with the exceptions mentioned above, the measured values are different from one test to another. In fact, the average value of  $q_{rem}/q_{in}$  is around 0.3 for the T-bar 2 cyclic tests while it is around 0.2 for T-bar 3. This comparison can be better illustrated when two tests performed at similar depths are plotted together, as in Fig. 23. Now, instead of plotting the normalized values, the absolute values for the same tests are compared in Fig. 24. It can be observed that there is a significant difference between the initial penetration values (cycle 0.5), 43 kPa or 27-37% of the initial penetration. However, the first extraction has reduced this difference to only 10 kPa (or 6-8%) of the initial value, and continued to reduce until the same values were found, at cycle 2.5. The values of  $q_{rem}$  of all tests (T-bar 2 and 3) are plotted vs. depth in Fig. 25, where it can be observed that except in two cases, all values are approxi-

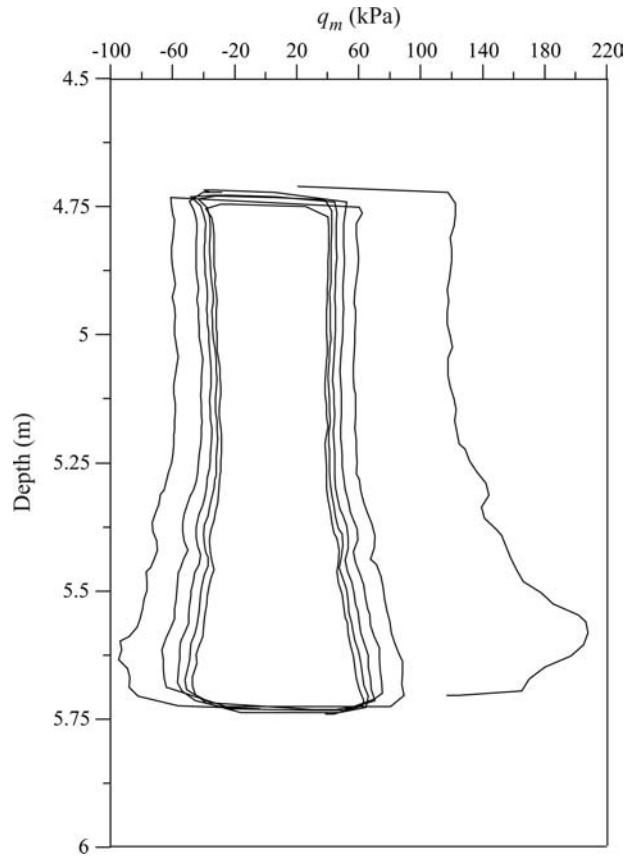


Figure 17 - T-bar 2-6 cyclic test.

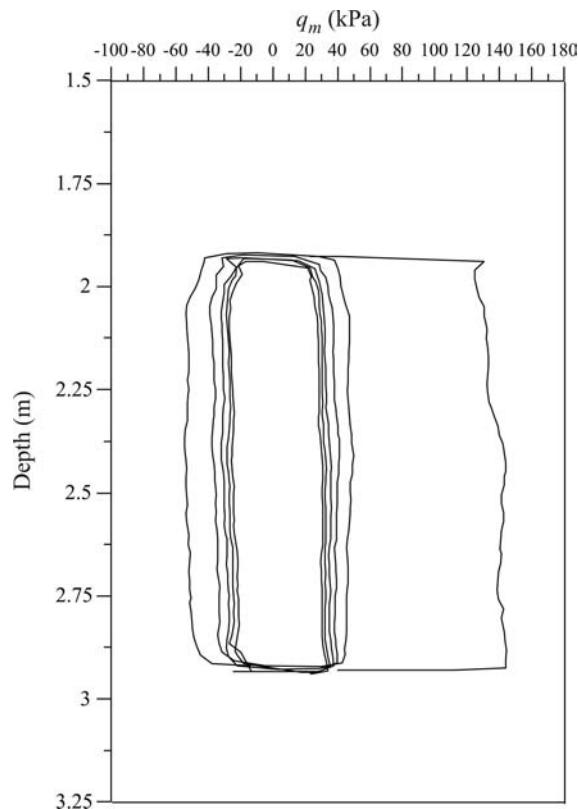


Figure 18 - T-bar 3-3 cyclic test.

mately the same, indicating that the influence of the roots have disappeared when the cycling procedure was applied.

Equation 7 was evaluated separately for cyclic tests performed at T-bar 2 and T-bar 3, and the corresponding results are found in Figs. 26 and 27, respectively. A significant difference can be observed from the results of the tests. Cyclic tests performed at T-bar 2 showed a trend very close to Eq. 7, except in the case of the tests performed at the smallest and greatest depths, due to the reasons previously discussed. The tests performed at T-bar 3 did not present the same results. In fact, the tests performed at similar depths at T-bar 3 are all apart from the curve representing Eq. 7, which is due to the  $q_{in}$  values being affected by the

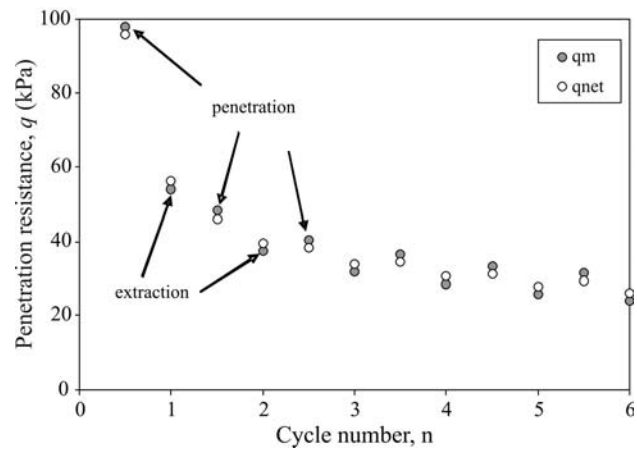


Figure 19 - Degradation curve, T-bar 2-3 cyclic test.

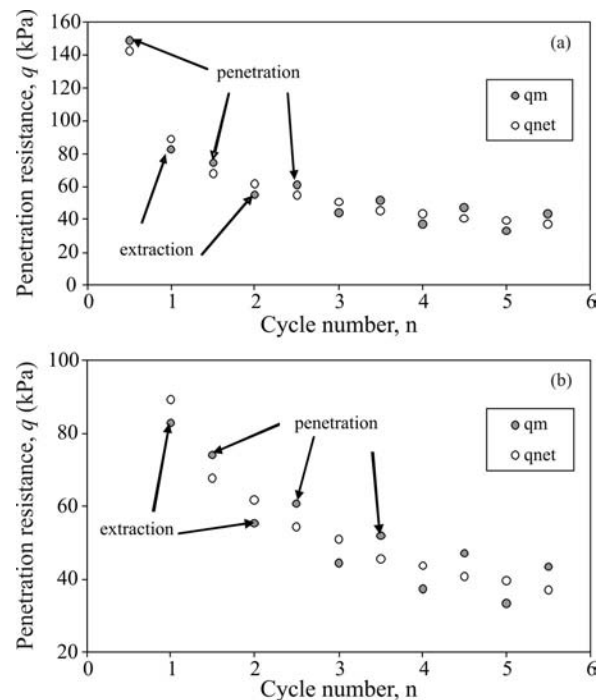


Figure 20 - Degradation curve, T-bar 2-7 cyclic test; (a) all data; (b) without initial penetration to allow magnification.

roots. Had this not occurred, similar results as for T-bar 2 would have been obtained, once  $q_{rem}$  values are about the same, as showed in Fig. 25. It can be concluded that Eq. 7 proved to be a useful tool to predict  $q_{rem}$  values based only in  $q_{in}$  and  $q_{ext}$  values in the case of Sarapu  II soft clay.

The whole degradation curve, predicted by Eq. 8, was compared with the data obtained from tests performed at T-bar 2 and 3, and are presented in Figs. 28 and 29, respectively. Cyclic test T-bar 2-3 is shown in Fig. 28, where a good matching between predicted and measured values has been obtained. All tests in T-bar 2 except T-bar 2-1 and 2-9, for the reasons discussed above, presented similar results.

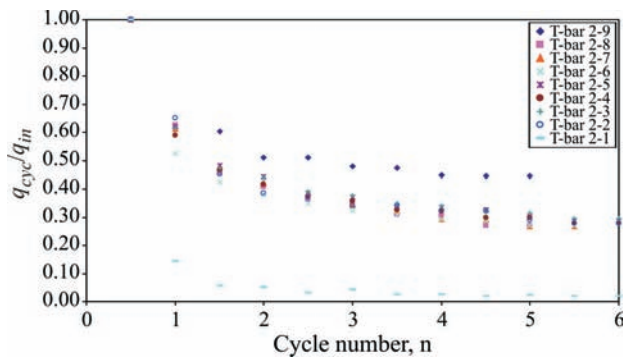


Figure 21 - Normalized cyclic degradation curves, T bar-2.

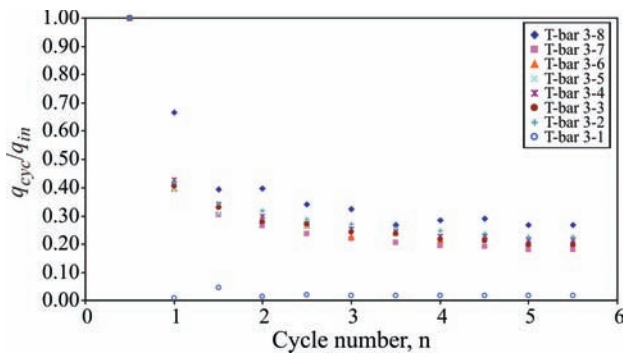


Figure 22 - Normalized cyclic degradation curves, T bar-3.

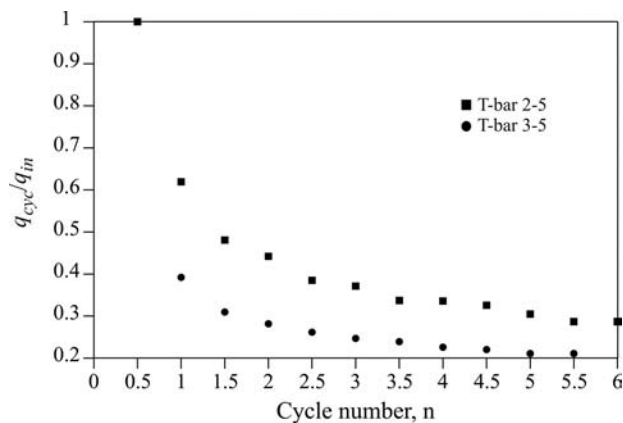


Figure 23 - Normalized degradation curves, T-bar 2-5 and T-bar 3-5 tests.

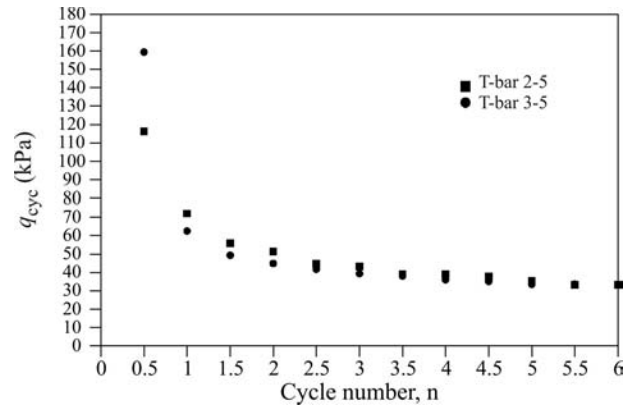


Figure 24 - Degradation curves, T-bar 2-5 and T-bar 3-5 tests.

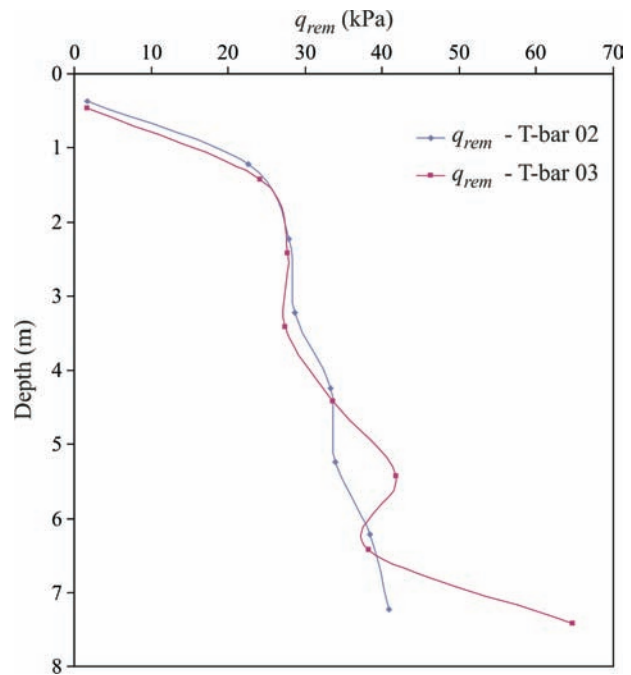


Figure 25 - Values of  $q_{rem}$  from all tests performed.

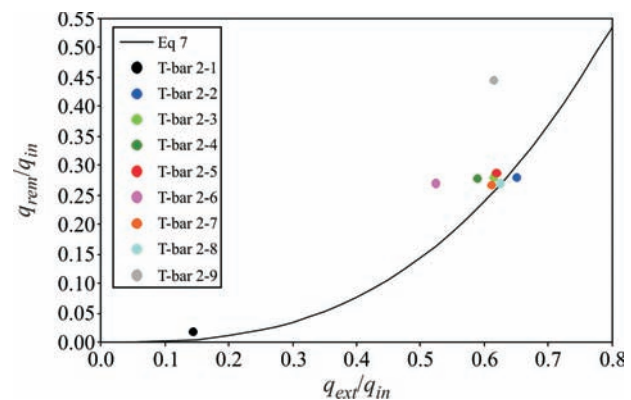
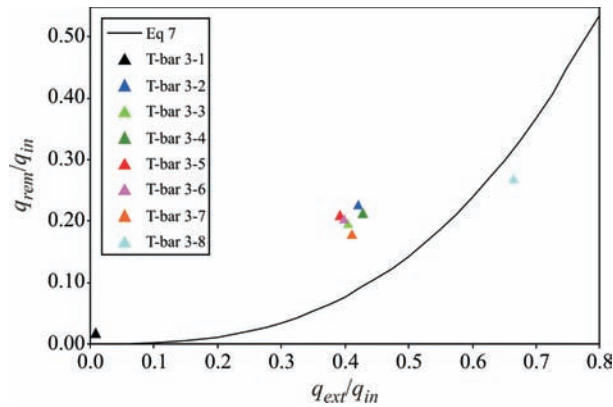
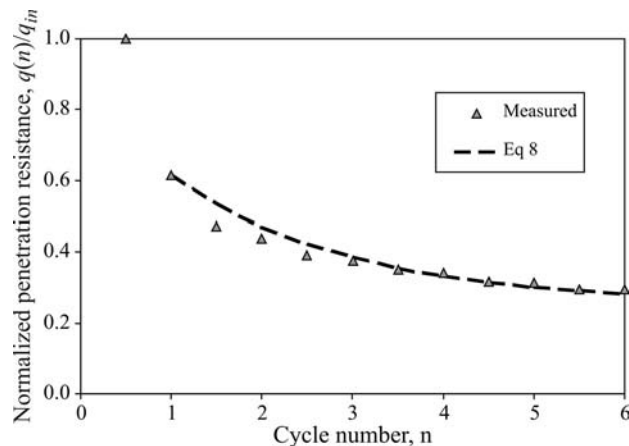


Figure 26 - Relationship between extraction ratio ( $q_{ext}/q_{in}$ ) and normalized remoulded resistance ( $q_{rem}/q_{in}$ ), data from T-bar 2 cyclic tests.

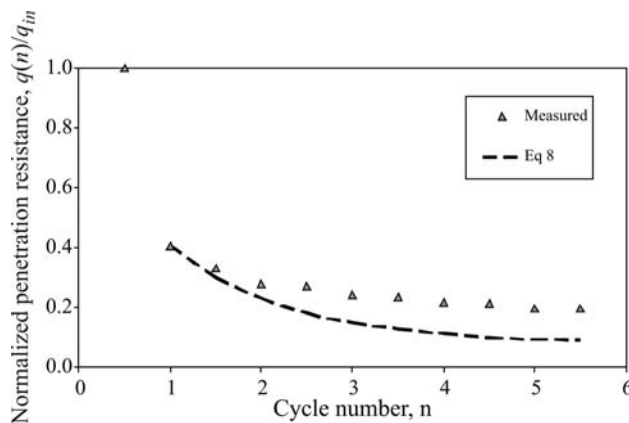
As far as T-bar 3 is concerned, a different trend was obtained. All tests presented a poor matching between predicted and measured values, as showed for test T-bar 3-3, which may be considered a typical test. This is an expected behaviour, since Eq. 8 is an empirical equation based on “well behaved” soils and regular conditions, *i.e.* the pres-



**Figure 27** - Relationship between extraction ratio ( $q_{ext}/q_{in}$ ) and normalized remoulded resistance ( $q_{rem}/q_{in}$ ), data from T-bar 3 cyclic tests.



**Figure 28** - Normalized cyclic degradation, T-bar 2-3.



**Figure 29** - Normalized cyclic degradation, T-bar 3-3.

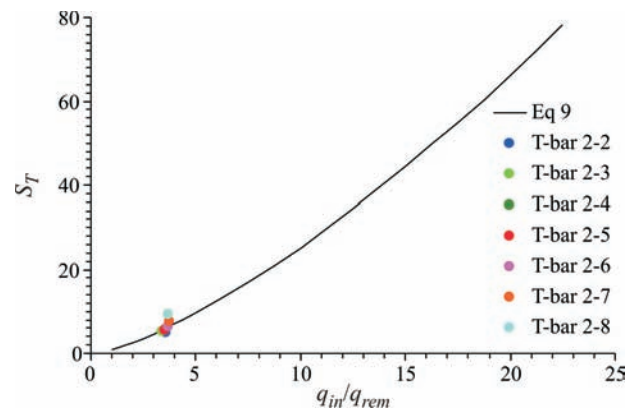
ence of roots influencing test results are not taken into account in the equation.

Equations 9 and 10 are represented in Figs. 30 and 31, where the experimental values are also included. The reference  $S_T$  values were obtained from the vane test results showed in Fig. 12b.

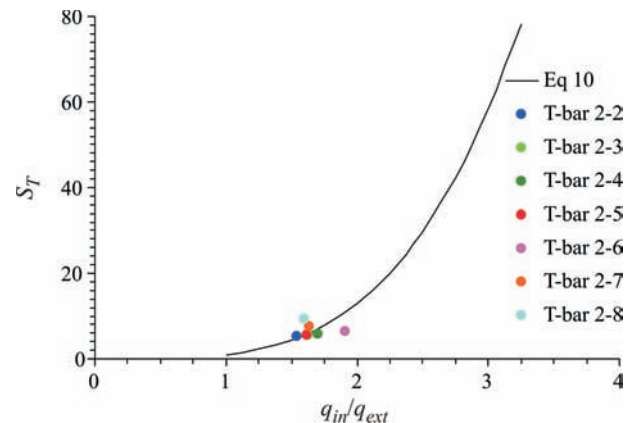
As expected, the cyclic tests corresponding to T-bar 2 have provided good results, and the tests related to T-bar 3 (not showed) have provided poor matching, for the reasons previously discussed.

As far as  $N_{T-bar}$  factors are concerned,  $N_{T-bar}$  obtained from tests not affected by the roots ranged from 8.8 to 10.9, with an average of 9.8, while  $N_{rem, T-bar}$  ranged from 14.1 to 19.5, with an average of 16.3. It must be pointed out that  $N_{rem, T-bar}$  were evaluated based on all cyclic T-bar tests, since the roots have not influenced the remoulded values.

Therefore  $N_{T-bar}$  values (related to the natural condition) were smaller than  $N_{rem, T-bar}$  (related to the remoulded condition). A possible explanation for this behavior was provided by Low *et al.* (2010), attributing it to the soil being partially remoulded during the initial penetration of the T-bar, whereas the soil becomes fully remoulded locally at the end of a cyclic test. As a result, the strength enhancement owing to high strain rate around the T-bar is partly



**Figure 30** - Sensitivity against  $q_{in}/q_{rem}$ , T-bar 2 cyclic tests.



**Figure 31** - Sensitivity against  $q_{in}/q_{ext}$ , T-bar 2 cyclic tests.

compensated by the strength reduction owing to partial remoulding during the initial penetration, but not after remoulding.

Moreover, there is a trend of  $N_{rem, T-bar}$  to increase with the increase of sensitivity, as showed in Fig. 32, as predicted from Eq. 12. It must be noted that all values - except those with different trends, previously discussed - were included, *i.e.* values from both T-bar 2 and 3 were included in the figure, since the remoulded values were not affected by the influence of the roots.

## 6. Additional Remarks and Conclusions

Seventeen T-bar cyclic tests in two deployments have been performed at Sarapu  II soft clay test site. In a third deployment only penetration was recorded.

The presence of roots has influenced the values of the initial penetration in one of the deployments, as observed in another test site, which is a consequence of the shape of the penetrometer and may be considered a shortcoming of the test. Therefore, to know whether roots have influenced the test results in a site at least two repeatable tests must be performed. If cyclic tests are performed, their results can provide a good indication of the influence of the roots.

The equations suggested by Yafrate *et al.* (2009) to evaluate the remoulded penetration resistance, the whole degradation curve and also the sensitivity, based only on the initial penetration and extraction resistances have provided good results for the Sarapu  II soft clay. The tests performed at very shallow depth (less than 1 m deep), however, did not provide good predictions in general, which was attributed to either: (i) the presence of roots, *i.e.* the value of  $q_m$  (initial penetration value) is mostly due to the resistance offered by the roots, which is not present when the cyclic test is performed; (ii) the very low effective stresses, which preclude the occurrence of the full-flow mechanism. More research is needed on this subject, since T-bar tests are very often used in the design of pipelines, where the values corresponding to very low depth are of paramount importance.

Considering the vane shear test as reference for obtaining  $s_u$  and  $s_{ur}$ ,  $N_{T-bar}$  obtained from tests not affected by

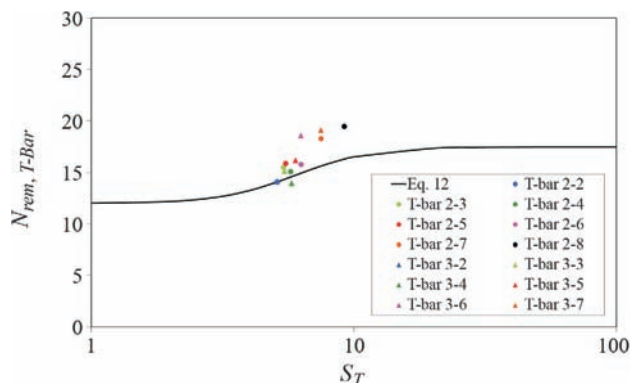


Figure 32 -  $N_{rem, T-bar}$  vs.  $S_T$ .

the roots ranged from 8.8 to 10.9, with an average of 9.8, while  $N_{rem, T-bar}$  ranged from 14.1 to 19.5, with an average of 16.3. Therefore  $N_{T-bar}$  values (related to the natural condition) were smaller than  $N_{rem, T-bar}$  (related to the remoulded condition).

## Acknowledgments

The authors would like to acknowledge the financial support provided by CENPES/PETROBRAS, and the allowance of Comando da Est     R     da Marinha no Rio de Janeiro to use part of its facilities at BR-040 motorway as a geotechnical test site.

Tom Lunne, from the Norwegian Geotechnical Institute (NGI), reviewed the paper and offered valuable comments.

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## ***Technical Note***

***Soils and Rocks***  
**v. 35, n. 3**



# A Short Review on the Importance of Colonnades, Entablatures and “Fault Joints” for the Excavation of Basaltic Rocks

G.R. Sadowski

**Abstract.** Entablatures, colonnades and “fault-joints” have been found in many excavations of dam foundations, tunnels and quarries in basaltic rocks of southern Brazil, but frequently they were not formally identified as such, and the two first, only rarely. This lack of recognition led many times to misunderstanding of their importance to slope stability and foundation problems. This short note reviews their basic definitions and stresses the influence of their properly consideration in the development of geotechnical projects.

**Keywords:** entablature, colonnade, basalt, jointing, columnar, dam.

## 1. Introduction

Entablatures, colonnades and “fault-joints” have been met during dam construction in Brazilian basalts but sometimes not identified as such. They may be frequently a concern in stability, excessive overbreak and seepage in dam foundations and also commonly in slope stability.

Entablatures and colonnades are widely known features in international scientific literature, but their description in Brazilian basalts has been done only after the studies of Souza Jr (1992). Even then, references were practically restricted to reports of quarry pit excavations, considerations about rock weathering susceptibility or concerns on the role they play in groundwater exploration (Fernandes *et al.*, 1998; Gomes & Rodrigues, 1999).

“Fault-joints” on the other hand were longer identified in Brazil but not described as such in international technical literature due probably to the preference in using the term “platy - joints”.

This short note reviews their basic definitions and stresses the influence of their proper consideration in the development of geotechnical projects.

## 2. Definitions

There are several geological structures in basaltic flows that belong to the common geologists jargon: spiracles, lava tubes, breccias, volcanic glasses, columnar joints, etc. (Cetin *et al.*, 2000). Other terms are less popular, like “maars” (Araujo *et al.*, 1977), “fault joints” (Bjoernberg & Kutner, 1987) and entablatures Souza Jr. (1994a, b).

### 2.1. Colonnades and entablatures

Colonnades and entablatures are features or tiers of columnar cooling joints that may occur jointly in a single

basalt flow (Fig. 1) giving sometimes “a false appearance of the existence of two flows” (Sherbon-Hills, 1976).

Colonnades are composed mainly by assemblages of relatively large diameter (0.3 to 2 m) and well-formed columns, frequently with pentagonal to hexagonal cross sections, that occur mainly in the lower or upper third of a flow. The columns are usually perpendicular to the base of the flow (presumably normal to the cooling isotherms).

Entablatures usually overlay the basal colonnade and show thinner and longer or slender columns, with a diameter of circa 0.2 to 0.5 m. They may be irregular to hackly, originating local radiating patterns, deviating from being perpendicular to the base of the flow (Fig. 1). Entablatures give frequently way to an upper colonnade, which, on its turn, is topped by vesicular basalt, but other recurrences are found in nature as described by Long & Wood (1986) (Fig. 2).

While colonnades are usually related by a somewhat slower cooling rate starting from the base to the top of the flow, entablatures have been related to faster downward cooling possibly related to quenching by flowing water from above (heavy rains or flooding). Entablatures usually show a larger (circa 40% more) percentage of fine matrix than colonnades.

In the field the separation between Entablatures and Colonnades is structurally sharp and, in certain cases, marked by a fault-joint. In what concerns the matrix of the rockmass the geomechanical differences, in our opinion, are still not clearly resolved (see, for example, Gomes & Rodrigues, 2008).

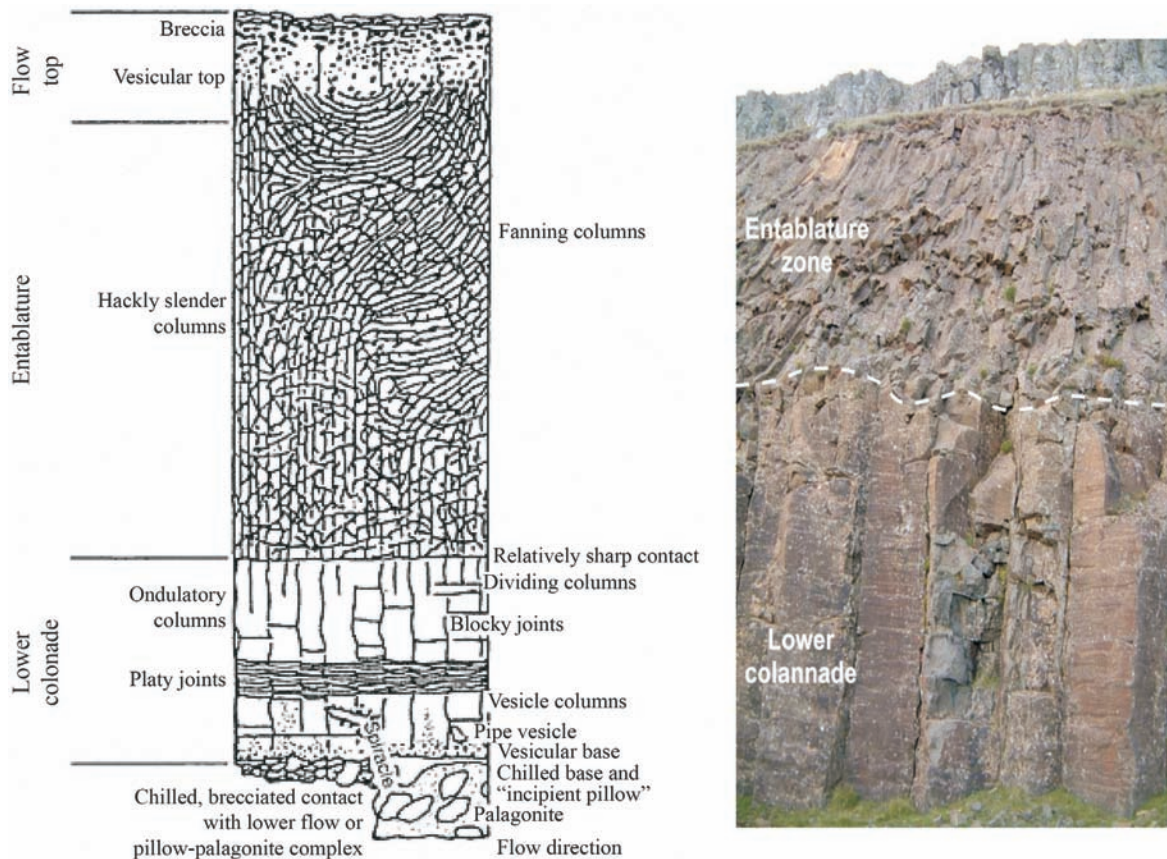
Since the matrix material from the entablatures is thinner and glassier it should be less porous than that from the colonnades and their texture therefore much more compact and the geomechanical properties of the rock masses

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Submitted on February 29, 2009; Final Acceptance on November 21, 2011; Discussion open until April 30, 2013.

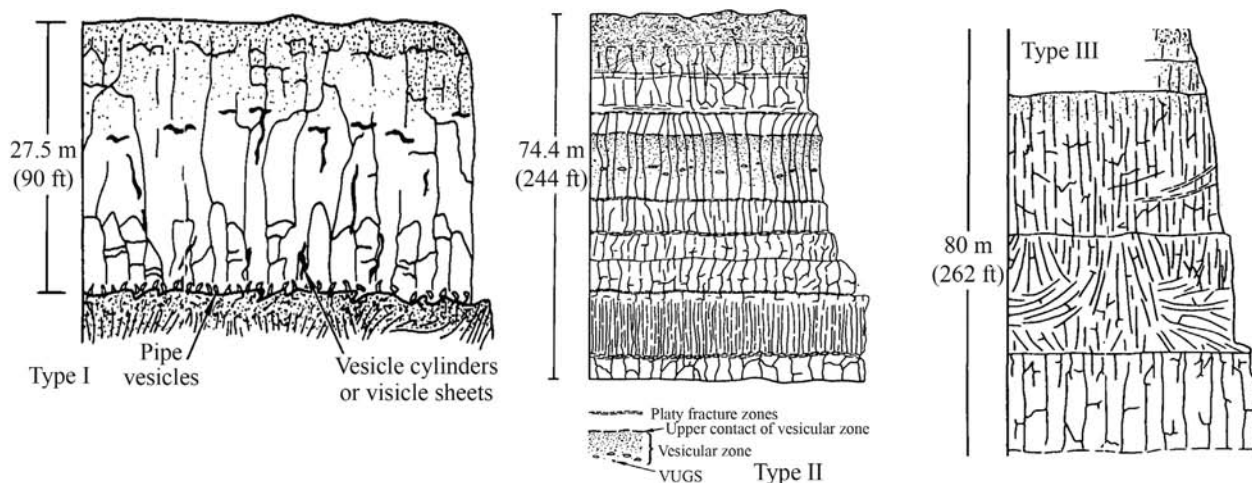
should vary accordingly. In the entablatures domain, the uniaxial compression strength and the elasticity modules are expected to be higher.

Structurally, jointing is denser in the entablatures domain, but joints seem to be partially welded or cemented by

thin films of minerals (Fig. 3). They are tightly closed in fresh rock and opened in weathered rock masses, where hackles turn to be more evident and help to induce fragmentation of the columns and thus, some raveling in open cuts (Fig. 4).



**Figure 1** - Intraflow structure of a basalt flow. Scheme on the left according to Swanson & Wright (1981).



**Figure 2** - Types of flows in terms of structural organization of cooling joints tiers (apud Long & Wood, 1986). Type I: flow - with no entablature, usually found in the Columbia River Basalts as related to relatively thin flows (10 to 20 m); Type II: flow: with alternating colonnades and entablatures, usually in flows with a thickness of 45 to 75 m; Type III: flow: with a lower colonnade and an upper entablature. Both, type I and II may have an upper colonnade.



**Figure 3** - Slender and partially welded columns.

## 2.2. “Fault-Joints”

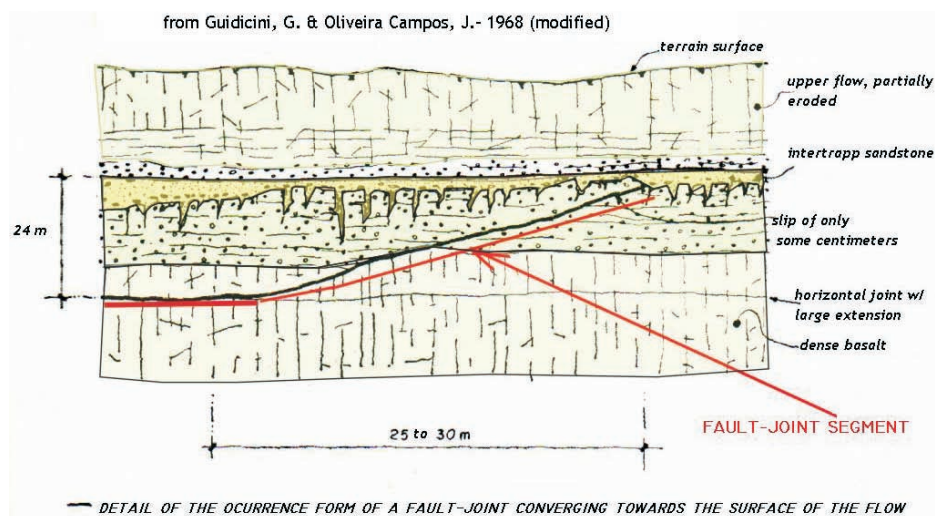
“Fault-Joints” were originally defined as such in Brazil by Guidicini & Oliveira (1968) (Fig. 5) at the Ibitinga Dam construction site, and successively quoted in other dam constructions. such as Foz de Areia, São Simão, Volta



**Figure 4** - Slope cut raveling in loosened entablature basalt.

Grande, Itaipu etc. in the basalt flows province of the Parana Basin (Bjoernberg & Kutner, op. cit., Souza Jr. & Oliveira Campos, 1987 and many others). One may probably relate them to the term “platy-joints” used in the anglo-saxon literature (Sherbon-Hills op. cit. and others).

These structures are usually represented by a planar sub-horizontal fracture or lamination surface which may gradually incline (dip) about  $35^\circ$  or more. It may be also braided or divided (moustache structure), sometimes ex-



**Figure 5** - Fault-joint as defined in Guidicini & Campos (1968).

hibiting striations which allowed some tectonic speculations. Their origin has been related to different mechanism such as differential contractions during cooling, stress relief effects or even shallow tectonics. Being relatively permeable, they are frequently percolated and affected by differential weathering. Consequently, the laminated platy rock of the densely jointed zone may be totally or partially weathered. Shear strength properties are usually substantially lower than those of the rest of the rock mass. Mohr-Coulomb strength parameters have been determined, some with the help of “in situ” shear tests, to spread frequently around  $\sim 0.05$  MPa for the cohesion and a  $\phi$  of around  $20$  to  $35^\circ$  depending on the type of filling or the presence of evidences of pre-shearing (Marchi & Cury Jr., 1983) (Fig. 6). Figures 7 and 8 show their aspect in other dam construction sites.

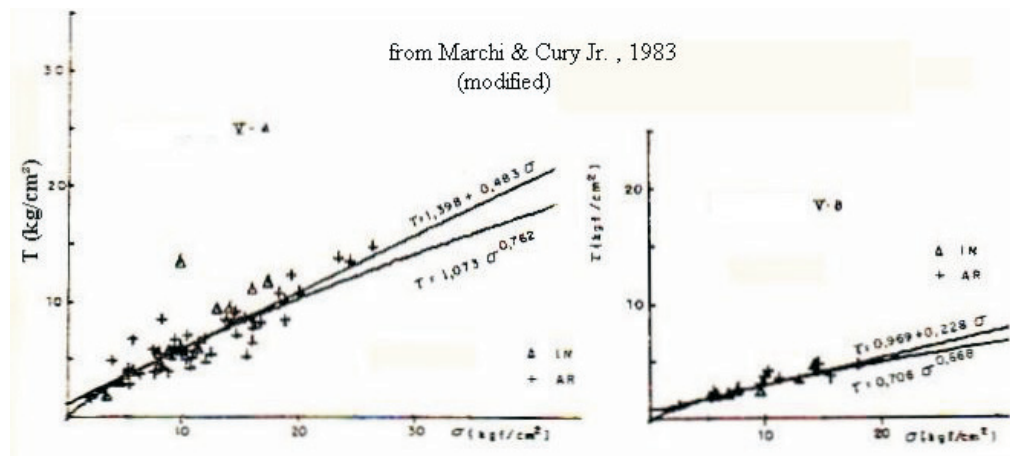
### 3. Conclusive Remarks: The Influence of these Features in the Development of Geotechnical Projects

Depending on the joint orientation, one may conclude that the presence of entablatures may lead to a higher direc-

tional permeability in dam foundations and to a higher potential of rock raveling at high cuts (as in Fig. 4). The raveling and the ease of excavation or even risk of over-excavation in the more superficial horizons with the use of machines has been observed, particularly once one breaks the small cohesion between the partially welded columns (Fig. 9). However in what concerns permeability or bearing strength of the mass, no special problematic behavior has been determined. Both aspects ask for careful blasting in order to achieve the desired design profile.

Concerning the production of raw material, the differences between colonnade and entablature are also very important. Although favoring the fast production of hand-size blocks, the larger percentage of glass found in entablatures increases the percentage of splinters or lamellar particles and results in the need of special cements to diminish the reactivity with concrete.

In what concerns fault-joints, problems related to their lower shear strength and higher permeability have been of common concern amid Brazilian engineers and geologists. Reinforcements through anchoring or indentations of the concrete structures founded above these frac-



**Figure 6** - Mohr Coulomb direct shear strength envelopes for sound and weathered fault joints (IN = intact, AR-after rupture) from Marchi & Cury Jr (1983).



**Figure 7** - “Fault-joints” in basalts of Northern Brazil. Observe ramifications on the left end. The photo was taken during dam construction.



**Figure 8** - Weathered “Fault-joint” between colonnade and entablature (upper realm) at a dam construction site in Parana Basin basalt flow in the São Paulo State.



**Figure 9** - Machine excavation of blocky, partially weathered entablature. São Paulo State.

tures may be needed. Grouting or even removal of the soft filling followed by its substitution by concrete are also some of the preventive measures that have been implemented.

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# The Influence of Moisture Content on Tensile and Compressive Strength of Artificially Cemented Sand

F. Stracke, J.G. Jung, E.P. Korf, N.C. Consoli

**Abstract.** Applying Portland cement to soils is an excellent technique when it is necessary to improve local soil for the construction of stabilized pavement bases and to have a support layer for shallow foundations. Consoli *et al.* (2007, 2009, 2010) developed a rational dosage methodology for artificially cemented soils based on porosity/cement index, which can be applied to unconfined compressive strength, as well as to splitting tensile strength. Furthermore, a unique  $q/q_u$  relationship was found, independent of the cement content and voids ratio. Following the assessment of the main factors that influence the strength of artificially cemented soils, the present research aims to quantify the influence of the moisture content in the tensile and compressive strength of an artificially cemented sand. A program of splitting tensile tests and unconfined compression tests was carried out. There were tested three voids ratio (0.65, 0.73 and 0.81), four cement contents (3%, 5%, 7% and 9%) and five moisture contents (6%, 8%, 10%, 12% and 14%). The results show that the reduction in moisture content of the compacted mixture increases both the tensile and compressive strengths. Furthermore, it has been shown that  $q/q_u$  relationship was kept constant, being independent of the porosity/cement ratio and the moisture content.

**Keywords:** tensile strength, compressive strength, soil-cement, compacted soils, moisture content.

## 1. Introduction

Portland cement is worldwide used in the improvement of local soils. Several studies have shown that soil-cement blends have a complex behavior that is affected by many factors, such as the amount of cement, the porosity and moisture content at the time of compaction (Clough *et al.*, 1981, Tatsuoka and Shibuya 1992, Huang and Airey 1998; Horpibulsuk *et al.*, 2003, Consoli *et al.*, 2003, 2006, 2007, 2009, 2010, Thomé *et al.*, 2005, Dalla Rosa *et al.*, 2008).

Consoli *et al.* (2007) developed the first rational dosage methodology for artificially cemented soils considering the porosity/cement ratio ( $\eta/C_v$ ), defined as the porosity of the compacted soil-cement mixture divided by the volumetric cement content, as an appropriate index to evaluate the unconfined compressive strength of soil-cement mixtures. Further studies (Consoli *et al.*, 2009, 2012) quantified the influence of the amount of cement and porosity on the initial shear modulus ( $G_0$ ) and effective strength parameters ( $c'$ ,  $\phi'$ ) of artificially cemented sandy soils.

Additionally, Consoli *et al.* (2010) showed that the porosity/cement ratio ( $\eta/C_v$ ) was an appropriate index to evaluate not only unconfined compressive strength ( $q_u$ ) of soil-cement mixtures, but splitting tensile strength ( $q_t$ ) as well. The  $q/q_u$  relationship was shown to be unique for a given soil-cement mixture studied, being independent of the porosity/cement ratio.

All these previous studies covered a wide range of voids ratio (corresponding to high, medium, and reduced relative densities) and of cement percentages. The only variable that was not assessed was the moisture content, which was the same in all studies ( $w = 10\%$ ). Based on this fact, there is still a variable (moisture content) that has to be better evaluated in soil-cement mixtures. Therefore, this study aims to quantify the influence of moisture content in the tensile strength ( $q_t$ ) and compressive strength ( $q_u$ ) of artificially cemented sand. Moreover, the variation of the  $q/q_u$  relationship with moisture content and porosity/cement ratio is evaluated.

## 2. Experimental Program

The experimental program has been carried out in two parts. First, the geotechnical properties of the soil and cement were characterized. Then, a number of splitting tensile and unconfined compression tests were carried out.

### 2.1. Materials

The soil used in the testing was a sand obtained from the region of Osório, near Porto Alegre, in Southern Brazil, classified (ASTM 1993) as non-plastic uniform fine sand (SP). The mean effective diameter ( $D_{50}$ ) of the studied sand is 0.16 mm, being the uniformity and curvature coefficients of 1.9 and 1.2, respectively. Sand particles have a rounded shape and specific gravity of the solids is 2.65. Mineralogical analysis showed that sand particles are predominantly

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Submitted on December 20, 2011; Final Acceptance on November 22, 2012; Discussion open until April 30, 2013.

quartz. The minimum and maximum void ratios are 0.6 and 0.9, respectively. The angle of shearing resistance at constant volume is about 30°.

High early strength Portland cement was used as the cementing agent. Its fast gain of strength allowed the adoption of seven days as the curing time. The specific gravity of the cement grains is 3.15. Distilled water was used for these characterization tests and for molding specimens for the tensile and compression tests.

## 2.2. Methods

### 2.2.1. Molding and curing of specimens

For the splitting tensile and unconfined compression tests, cylindrical specimens 50 mm in diameter and 100-mm high were used. Once established a given voids ratio ( $e$ ), the target dry unit weight ( $\gamma_d$ ) was calculated according to Eq. 1

$$e = \frac{\gamma_s}{\gamma_d} - 1 \quad (1)$$

where  $\gamma_s$  = solids unit weight. A target dry unit weight for a given specimen was then established through the dry mass of soil-cement divided by the total volume of the specimen. In order to keep the dry unit weight of the specimens constant with increasing cement content, a small portion of the soil was replaced by cement. As the specific gravity of the cement grains (3.15) is greater than the specific gravity of the sand grains (2.65), for the calculation of void ratio and porosity, a composite specific gravity based on the sand and cement percentages in the specimens was used.

After the sand, cement, and water were weighed, the sand and cement were mixed until the mixture acquired a uniform consistency. The water was then added continuing the mixture process until a homogeneous paste was created. The amount of cement for each mixture was calculated based on the mass of dry sand and the moisture content. The specimen was then statically compacted in three layers inside a cylindrical split mold, which was lubricated, so that each layer reached the specified dry unit weight. The top of each layer was slightly scarified. After the molding process, the specimen was immediately extracted from the split mold and its weight, diameter, and height measured with accuracies of about 0.01 g and 0.1 mm, respectively. The specimens were then placed inside plastic bags to avoid significant variations of moisture content. They were cured for 6 days in a humid room at  $23^\circ \pm 2^\circ \text{C}$  and relative humidity of above 95%.

The samples were considered suitable for testing if they met the following tolerances:

- *Dry unit weight* ( $\gamma_d$ ): degree of compaction between 99 and 101% (the degree of compaction being defined as the value obtained in the molding process divided by the target value of  $\gamma_d$ );

and

- *Dimensions* : diameter to within  $\pm 0.5$  mm and height of  $\pm 1$  mm.

### 2.2.2. Splitting tensile tests

Splitting tensile tests followed Brazilian standard NBR 7222 (Brazilian Standard Association 1983). An automatic loading machine, with maximum capacity of 50 kN and proving rings with capacities of 10 and 50 kN and resolutions of 0.005 and 0.023 kN, respectively, were used for the unconfined compression tests.

After curing, the specimens were submerged in a water tank for 24 h for saturation to minimize suction. The water temperature was controlled and maintained at  $23 \pm 3^\circ \text{C}$ . Immediately before the test, the specimens were removed from the tank and dried superficially with an absorbent cloth. Then, the splitting tensile test was carried out and the maximum load recorded.

### 2.2.3. Unconfined compression tests

Unconfined compression tests have been systematically used in most experimental programs reported in the literature in order to verify the effectiveness of the stabilization with cement or to access the importance of influencing factors on the strength of cemented soils. One of the reasons for this is the accumulated experience with this kind of test for concrete. The tests usually followed Brazilian standard NBR 5739 (Brazilian Standard Association 1980), being simple and fast, while reliable and cheap.

The automatic loading machine and proving rings were the same used for the splitting tensile tests. Curing of specimens and acceptance criteria were exactly the same as for splitting tensile tests.

### 2.2.4. Program of splitting tensile and unconfined compression tests

The splitting tensile and unconfined compression tests constituted the main part of this research. The program was conceived in such a way as to evaluate, separately, the influences of the moisture content, cement content, porosity, and porosity/cement ratio on the mechanical strength of the artificially cemented sand.

The molding points were chosen considering moisture contents of 6, 8, 10, 12 e 14%, and voids ratio of 0.65, 0.73, and 0.81 (corresponding, respectively, to high, medium, and reduced relative densities). Each point was molded with four different cement percentages: 3, 5, 7, e 9%. These percentages were chosen following Brazilian and international experience with soil-cement (*e.g.*, Mitchell 1981; Consoli *et al.*, 2003, 2006, 2007, 2009, 2010, 2012; Thomé *et al.*, 2005). Two specimens were tested for each point for both splitting tensile and unconfined compression tests.

### 3. Results and Discussion

#### 3.1. Effect of moisture content on tensile and compressive strengths

The splitting tensile strength is a function of the porosity/cement ratio ( $\eta/C_{iv}$ ) for the five moisture content used (6, 8, 10, 12 e 14%). The porosity/cement ratio is expressed as porosity ( $\eta$ ) divided by the volumetric cement content ( $C_{iv}$ ), the latter expressed as a percentage of cement volume regarding total volume, defined by Eq. 2.

$$\frac{\eta}{C_{iv}} = \frac{\left(\frac{V_v}{V_{total}}\right)}{\left(\frac{V_c}{V_{total}}\right)} = \frac{V_v}{V_c} \quad (2)$$

where  $V_v$  = volume of voids (water + air) of the specimen;  $V_c$  = volume of cement of the specimen; and  $V_{total}$  = total volume of the specimen.

Figure 1 presents the correlation between porosity/cement ratio ( $\eta/C_{iv}$ ) and the splitting tensile strength ( $q_t$ ) of the

studied sand-cement mixes with moisture contents of 6, 8, 10, 12 and 14% [see Eq. 3 to Eq. 7, respectively].

$$q_t \text{ (kPa)} = 17623 \cdot \left(\frac{\eta}{C_{iv}}\right)^{-1.70} \quad (3)$$

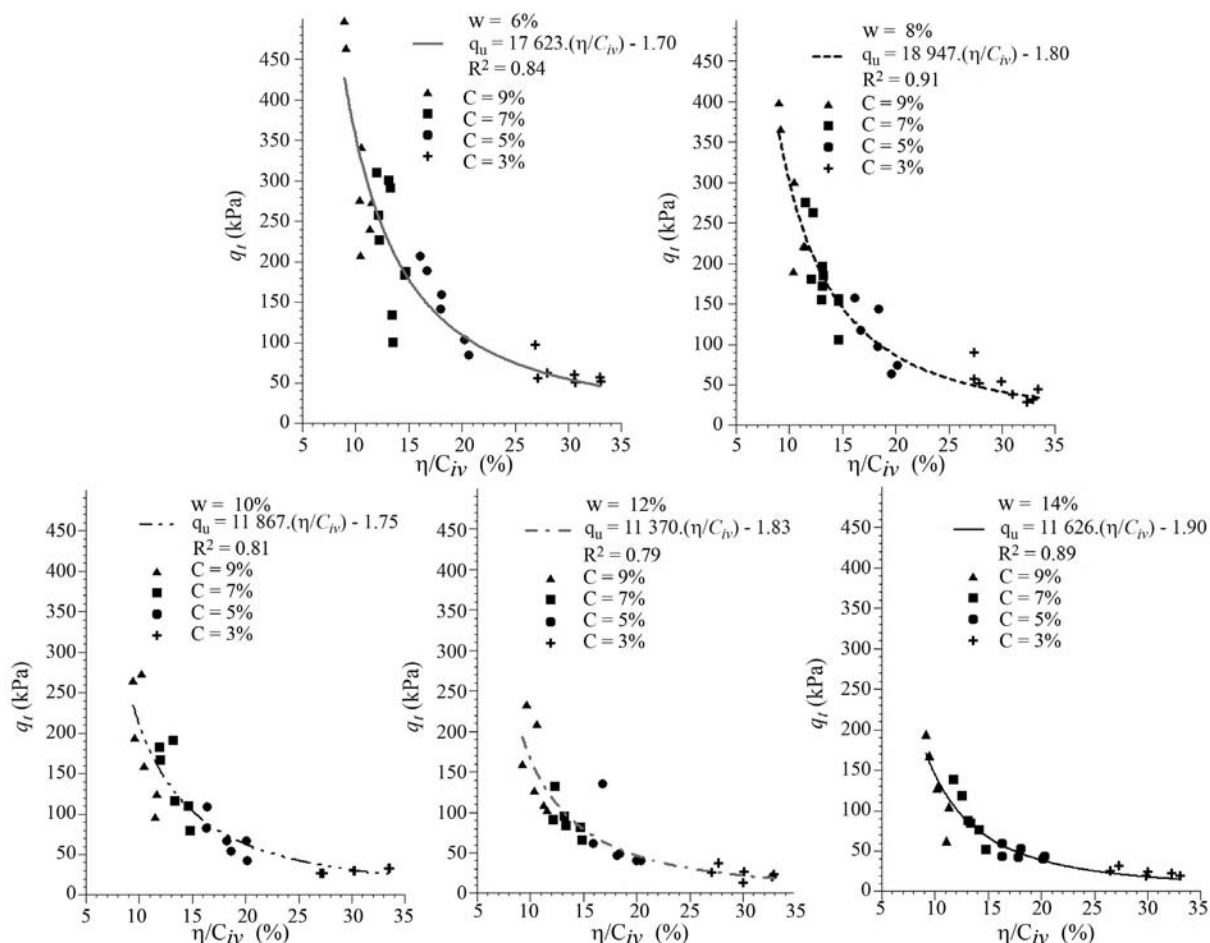
$$q_t \text{ (kPa)} = 18947 \cdot \left(\frac{\eta}{C_{iv}}\right)^{-1.80} \quad (4)$$

$$q_t \text{ (kPa)} = 11867 \cdot \left(\frac{\eta}{C_{iv}}\right)^{-1.75} \quad (5)$$

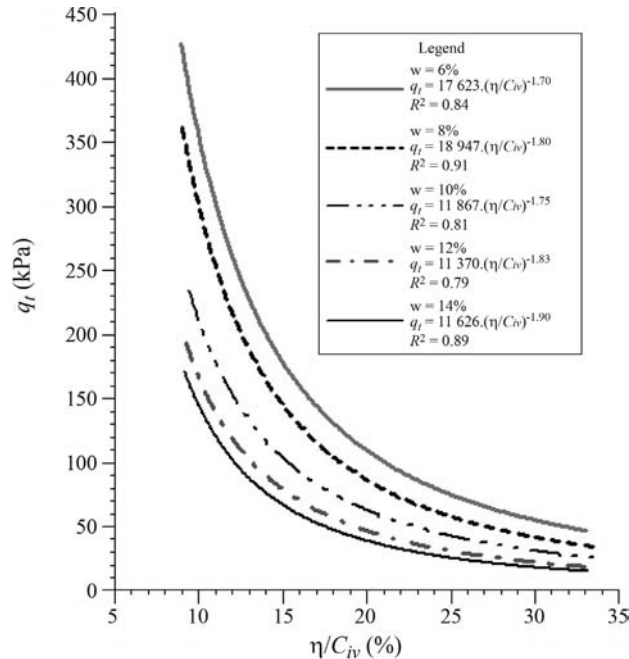
$$q_t \text{ (kPa)} = 11370 \cdot \left(\frac{\eta}{C_{iv}}\right)^{-1.83} \quad (6)$$

$$q_t \text{ (kPa)} = 11626 \cdot \left(\frac{\eta}{C_{iv}}\right)^{-1.90} \quad (7)$$

Figure 2 summarizes all results of splitting tensile strength, for all the moisture content tested (6, 8, 10, 12, 14%). In this figure, all the original exponents of the trend curves were kept. By examining this figure, it can be seen



**Figure 1** - Variation of splitting tensile strength ( $q_t$ ) with porosity/cement ratio for moisture contents of 6, 8, 10, 12 and 14%.



**Figure 2** - Variation of splitting tensile strength for the distinct moisture contents of the compacted mixture.

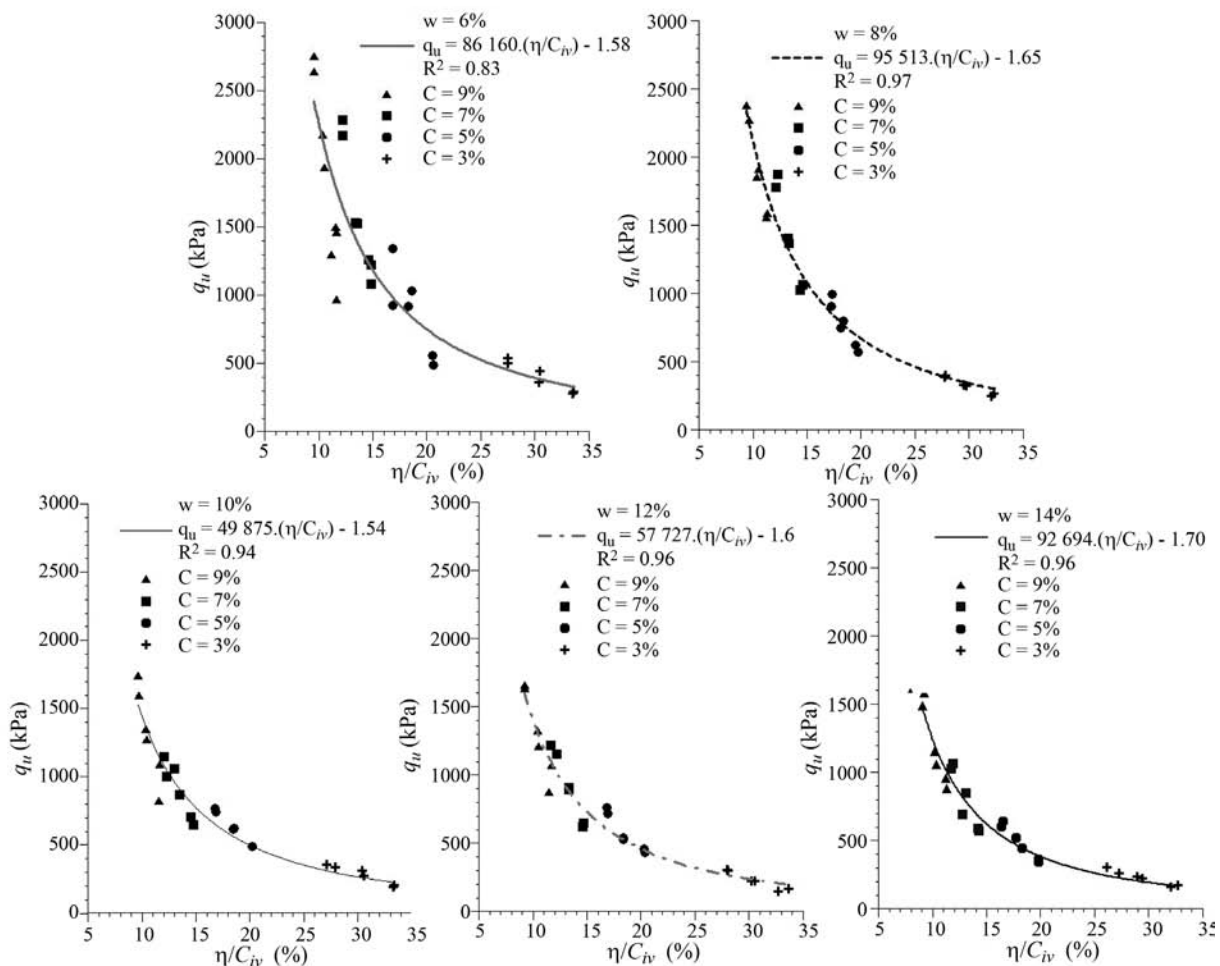
the influence of moisture content in the splitting tensile strength. The reduction in moisture content of compacted mixture increases splitting tensile strength. The possible explanation for such results is that the cement containing less water during mixture will have fewer pores in the cement-water mixture, ending in stronger cementitious bonds.

Figure 3 presents the correlation between porosity/cement ratio ( $\eta/C_{iv}$ ) and the unconfined compressive strength ( $q_u$ ) of the studied sand-cement mixtures with moisture contents of 6, 8, 10, 12 and 14% [see Eq. 8 to Eq. 12, respectively].

$$q_u \text{ (kPa)} = 86160 \cdot \left( \frac{\eta}{C_{iv}} \right)^{-1.58} \quad (8)$$

$$q_u \text{ (kPa)} = 95513 \cdot \left( \frac{\eta}{C_{iv}} \right)^{-1.65} \quad (9)$$

$$q_u \text{ (kPa)} = 49875 \cdot \left( \frac{\eta}{C_{iv}} \right)^{-1.54} \quad (10)$$



**Figure 3** - Variation of unconfined compressive strength ( $q_u$ ) with porosity/cement ratio for moisture contents of 6, 8, 10, 12 and 14%.

$$q_u \text{ (kPa)} = 57\,727 \cdot \left( \frac{\eta}{C_{iv}} \right)^{-1.61} \quad (11)$$

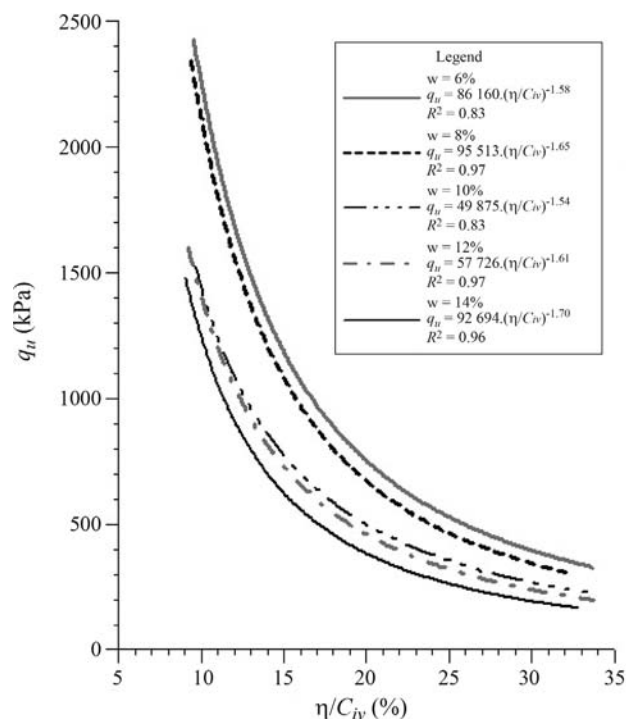
$$q_u \text{ (kPa)} = 92\,694 \cdot \left( \frac{\eta}{C_{iv}} \right)^{-1.70} \quad (12)$$

Figure 4 summarizes all the results of unconfined compressive strength tests, for all the moisture contents tested (6, 8, 10, 12, 14%). Also in this figure, all the original exponents of the trend curves were kept. By examining this figure, and equations in the graphic, it can be seen the influence of moisture content in the unconfined compressive strength. In the same trend of the results of splitting tensile strength tests, the reduction in moisture content of compacted mixture increases unconfined compressive strength.

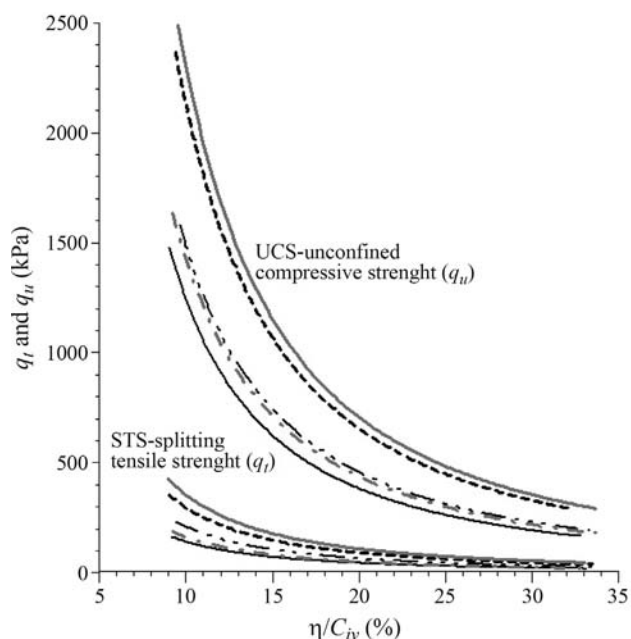
The results of present research show that moisture content of the sand-cement compacted mixtures influence the final tensile and compressive strengths. The reduction in moisture content of the compacted mixtures increases both the tensile and compressive strengths, as summarizes Figure 5.

Figure 5 shows all the exponential trend curves, with all the exponents adjusted to the average exponent (1.70). In this way, it is possible to compare different trend curves of different moisture contents.

Splitting tensile and unconfined compressive test results show that after 2% decrease in the moisture content there is an average increase of 17% in tensile and compressive strengths.



**Figure 4** - Variation of unconfined compressive strength for the distinct moisture contents of the compacted mixture.



**Figure 5** - Variation of unconfined compressive strength ( $q_u$ ) and splitting tensile strength ( $q_t$ ) for different moisture contents of the compacted sand-cement mixtures.

Furthermore, it can be concluded that splitting tensile strength - unconfined compressive strength ratio ( $q_t/q_u$ ) is about 13% for all moisture contents tested. These results corroborate the results obtained by Consoli *et al.* (2010), where  $q_t/q_u$  ratio was a constant for the sand-cement mixtures studied. Present study and the one developed by Consoli *et al.* (2010) showed that the  $q_t/q_u$  ratio is independent of both porosity/cement ratio and moisture contents. This is very interesting, in the sense that dosage methodologies based on rational criteria can concentrate either on tensile or compression tests, once they are totally interdependent.

#### 4. Conclusions

From the data presented in this technical note, the following conclusions can be drawn:

1. The reduction in moisture content of the compacted mixture increases both the tensile and compressive strengths.

2. A decrease of 2% in the moisture content increases both unconfined compressive strength and splitting tensile strength in about 17%, for the sand-cement mixtures evaluated in present research;
3. The  $q/q_u$  ratio is a scalar (0.13) for the sand-cement mixtures evaluated in the present study, being independent of moisture content and of porosity/cement ratio.

## Acknowledgments

The authors wish to express their gratitude to Brazilian Research Council (CNPq) for the financial support to the research group.

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## Notation

- $C$  = cement content (expressed in relation to mass of dry soil)
- $C_{iv}$  = volumetric cement content (expressed in relation to the total specimen volume)
- $D_{50}$  = mean effective diameter
- $e$  = voids ratio
- $q_t$  = splitting tensile strength
- $q_u$  = unconfined compressive strength
- $R^2$  = coefficient of determination
- $\gamma_d$  = dry unit weight
- $\gamma_s$  = solids unit weight
- $\eta$  = porosity
- $\eta/C_{iv}$  = porosity/cement ratio
- $\omega$  = moisture content

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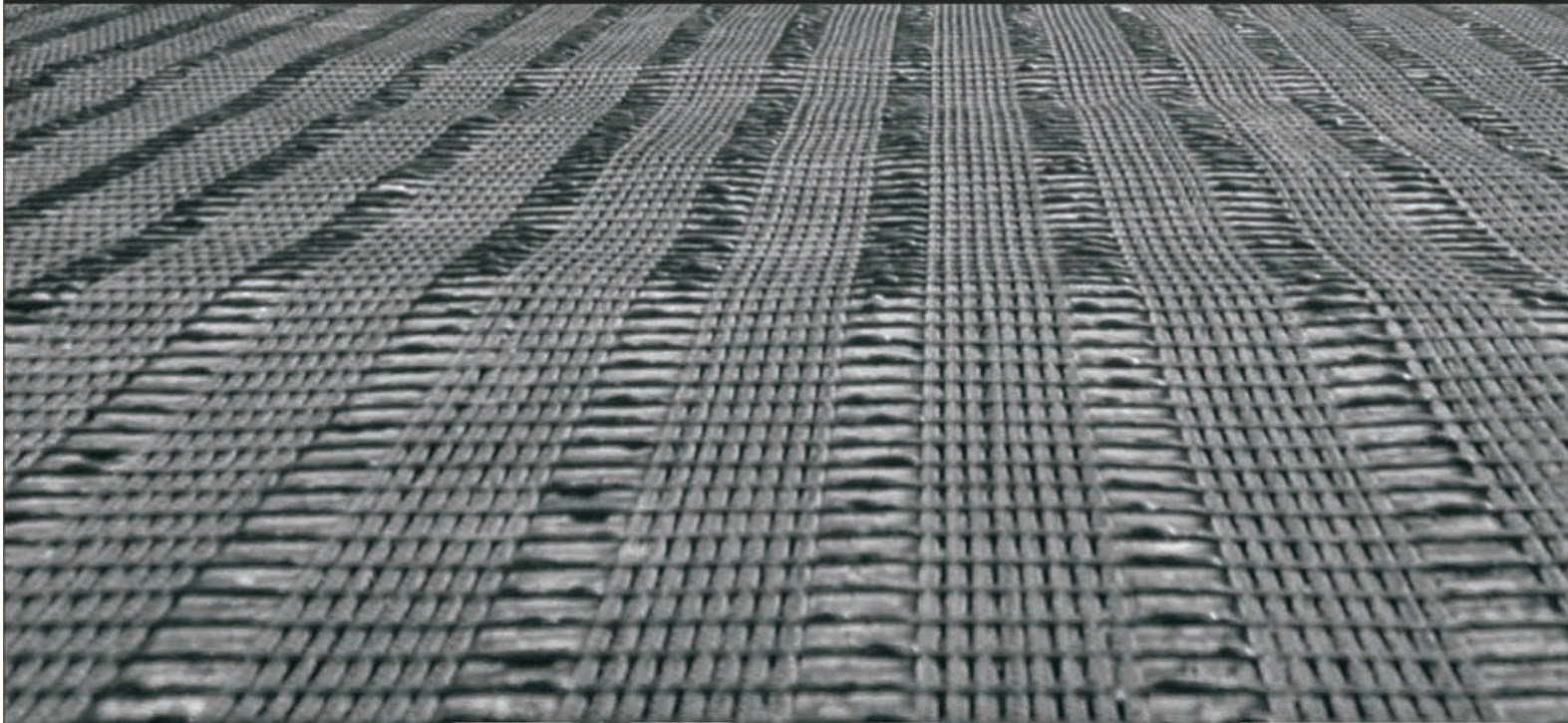
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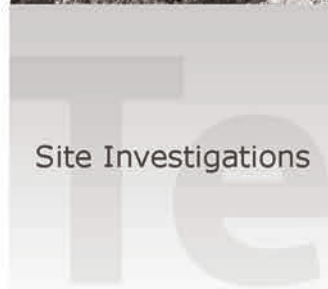
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Proceedings (printed matter or CD-ROM): Jamiolkowski, M.; Ladd, C.C.; Germaine, J.T. & Lancellotta, R. (1985) *New developments in field and laboratory testing of soils*. Proc. 11<sup>th</sup> Int. Conf. on Soil Mech. and Found. Engrg., ISSMFE, San Francisco, v. 1, pp. 57-153. (specify if CD - ROM)

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