

## Mechanical characterization of new micro-composites composed by natural clay matrix and PEG 6000 fillers

Mostapha El Jai<sup>1</sup>, Iatimad Akhrif<sup>2,4</sup>, Laila Mesrar<sup>3</sup>, Raouf Jabrane<sup>4</sup>

<sup>1</sup>Euromed School of Civil Engineering, Euromediterranean University of Fez, Morocco

<sup>2</sup>Laboratoire Systèmes et Environnements Durables (LSED), Faculté des Sciences de l'Ingénieur (FSI), Université Privée de Fès (UPF), Fez, Morocco

<sup>3</sup>LOMC, Le Havre University, France

<sup>4</sup>Geosciences and Environment Laboratory, Science and Technique Faculty, Sidi Mohamed Ben Abdellah University, Fez, Morocco

m.eljai@ueuromed.org

**Abstract.** The aim of this paper is to characterize mechanically the new micro-composites that have been developed in our laboratories. The composites are composed by natural clay (as a matrix) with variant percentages of Polyethylene Glycol 6000 (PEG 6000) as micro-fillers. We used the compression test for the measurement of the static parameters such as elasticity modulus in elastic region and the hardening coefficient which permits to describe the plasticity behaviour of the materials. An additional energetic approach is proposed in order to quantify the evolution of the plasticity of the reinforced materials, caused by the PEG 6000, for different percentages of this polymer.

**Key-words:** Microcomposite/clay matrix/PolyEthylene Glycol 6000, Mechanical properties, Elasticity, Plasticity, energetic analysis.

### 1. Introduction

This work is the third step of a large study that have been started by the elaboration of new micro-composites made by clay matrix and the addition of the PEG 6000, in order to valorize the mechanical, geotechnical and thermal properties of the original clay material of Fez region, Morocco. In a previous works (Akhrif & al. 2014, 2015 and 2016), we have presented and discussed the elaboration protocols of the materials, the microstructural transformations according to XRD spectra, and thermal analysis according to the DSC characterization.

The aim of this article is to quantify the mechanical behavior according to compression tests. The characterization was performed on cylinders that are presented in the next sections. The findings correspond to Young modulus, the mechanical strength and the loss of mechanical energy due to plastification of the materials.

### 2. Material and methods

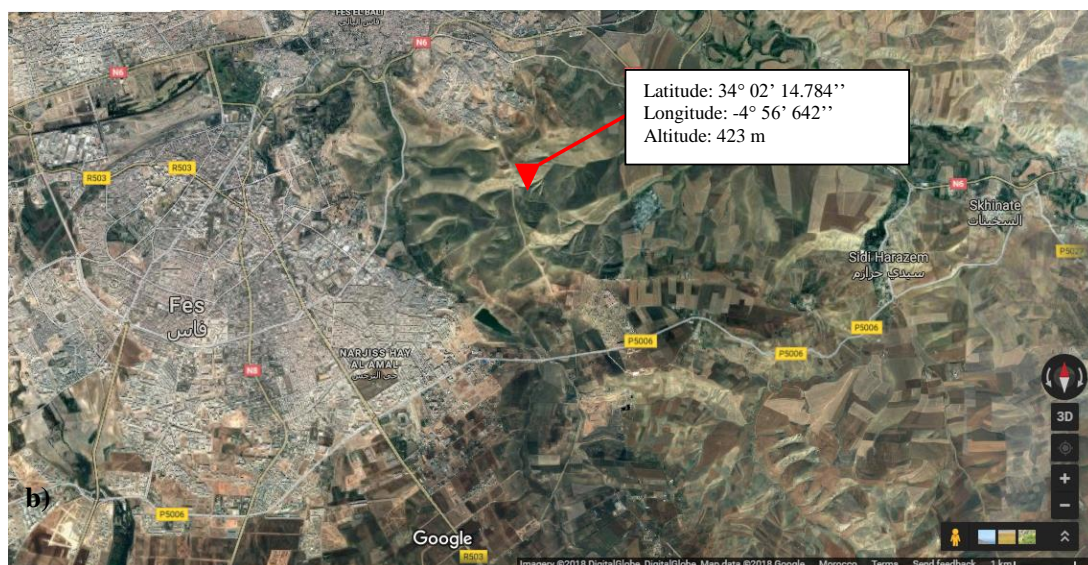
#### 2.1. Materials

##### 2.1.1. Natural clay used

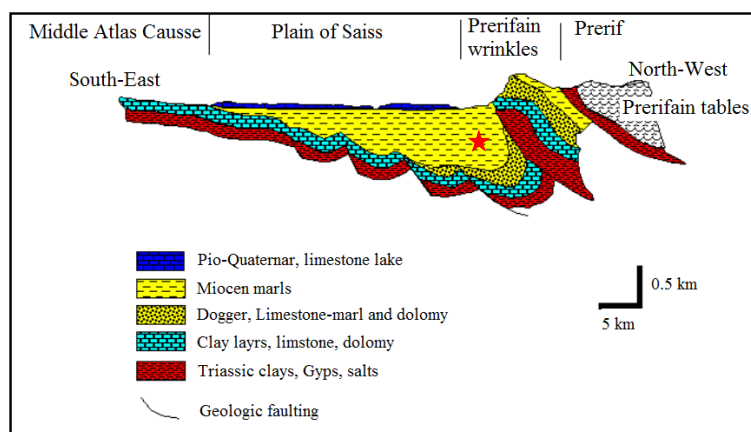


The clay matrix used corresponds to natural marl/clay from Ben Jellik quarry localized in the east of Fez city as shown in figure 1. Figure 1 shows also the Lambert coordinates of the region that correspond to “Longitude: 34°02’14.784” – Latitude: -4°56’56.642” – Altitude: 431 m”. A lithographic section, given in figure 2 permits to see the geologic formation of the extraction region.

The clay used corresponds to the Miocene marl, shown on figure 2 by a red star, extracted on a depth of 20 m. According to XRD analysis [1] [4], the material is composed by the clay phases illite and Kaolinite, calcite and quartz essentially.



**Fig. 1.** Localization of the extraction site (Longitude: 34°02’14.784” – Latitude: -4°56’56.642” – Altitude: 431 m)



**Fig. 2.** Lithographic section of the clay source: extraction shown by a red star [1]

### 2.1.2. Polyethylene Glycole 6000

The polyethylene Glycole 6000 belongs to the polyethylene oxide class with a molar mass of 6000 g/mole. This is the simplest of the hydro-soluble polymer that exists [2]. The PEG 6000 is ecologic, biodegradable and biocompatible polymer [3]. The chemical structure is  $-(CH_2-CH_2-O-)_n$ . this material is semi-crystalline having a crystallinity ratio between 75% and 85% [2].

## 2.2. Methods

### 2.2.1. Elaboration of the materials

The microcomposites developed were obtained by a succession of grindings from the initial quarry rock, to the fine elements. The PEG 6000 has been included with the finest clay within the electrical Retsch RM 200 mortar [5] for 15 minute of simultaneous mixing and grinding.



**Fig. 3.** The grinder used to mixing Clay matrix and PEG 6000 [4]

Thereafter, the compression cylinders were processed using a metallic mold. The initial diameter of each cylinder was about 25 mm, and the initial height was about 30 mm. Shrinking phenomenon was described and modeled in [4] and [6]. The cylinders were after polished and destroyed in the compression test machine.

#### 2.2.2. Material processing parameters

The materials processing corresponds to a complete experiments plan, according to:

- 3 levels of grinding sizing, denoted 3, 5 and 7 (the finest size), related to the grinder scales;
- 6 levels of PEG 6000 percentages per 6g of clay material: 0.1g, 0.2g, to 0.6g.

Thus, the total of the combinations corresponds to 18, in addition with the compression test applied to the row materials (clay) according to the 3 different sizing. To ensure the repetitiveness of the test, we adopt 2 samples by each composition. The total compression tests correspond to:

$$N = (6+1) \times 3 \times 2 = 42 \text{ compression tests}$$

#### 2.2.3. Compression test procedure

The compression tests were performed using “EM 550-DeltaLab machine” with a maximal strength of 50 kN. The compression parameters are:

- Static compression, where the superior plate speed is 0.2 mm/s; the inferior plate is blocked;
- Force and displacement acquisition time step: 0.5 s;
- Ambient temperature.

### 3. Results

#### 3.1. Mechanical properties

The first findings correspond to the mechanical properties: Young modulus “E”, mechanical strength “R<sub>m</sub>” and plasticity coefficient known as hardening coefficient “n”, according to the follow models [7]:

- Elastic domain modelled by Hook law :

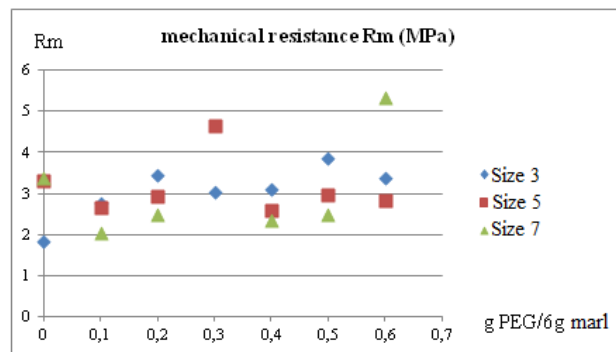
$$\sigma = E \varepsilon \quad (1)$$

- Plastic domain modelled by Ludwig law :

$$\sigma = \sigma_0 + k \varepsilon^n \quad (2)$$

##### 3.1.1. Mechanical strength

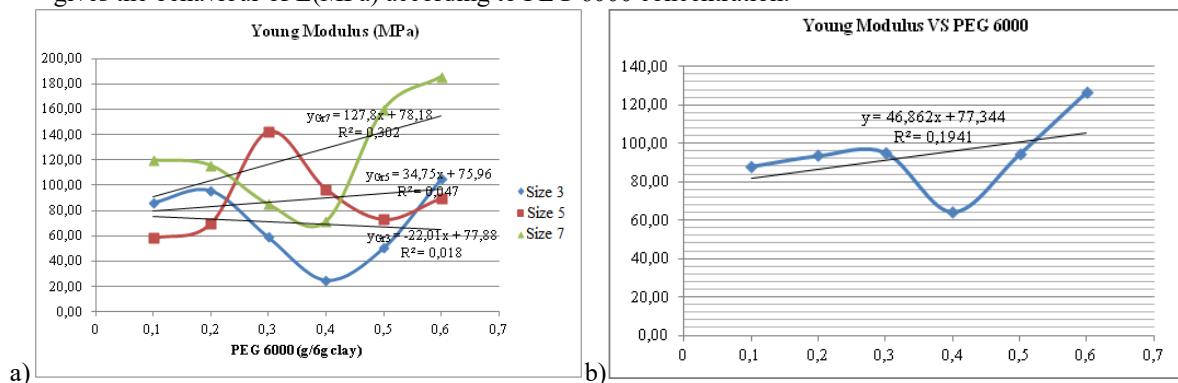
The mechanical strength corresponds to the maximum of stress  $\sigma$ . Figure 4 presents the distribution of the mechanical strength of the materials according to the two parameters: grinding size and the polymer concentrations. We observe that there is no significant evolution of R<sub>m</sub> with the two parameters. The values of R<sub>m</sub> oscillate around its initial values corresponding to the row clay material related to 0% of PEG.



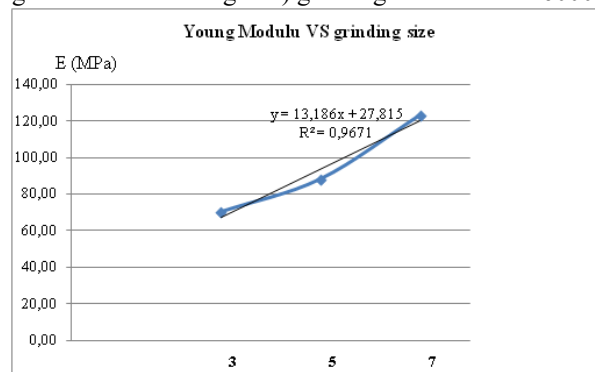
**Fig. 4.** Mechanical strength of the materials according to grinding size and PEG 6000 concentration

### 3.1.2. Young modulus

On figure 5-a, we can see that the Young modulus oscillates around an average value, and there is no significant evolution according to the evolution parameters. For each grinding size, we fit a regression line. The linear regression coefficient  $R^2$  calculated is very low, so the linear modelling is not accepted. Figure 5-b plot the average of the Young modulus according to clays size; thus, the curve gives the behaviour of  $E$ (MPa) according to PEG 6000 concentration.



**Fig. 5.** Young Modulus according to a) grinding size and PEG 6000 b) PEG 6000



**Fig. 6.** Young Modulus VS Grinding scale

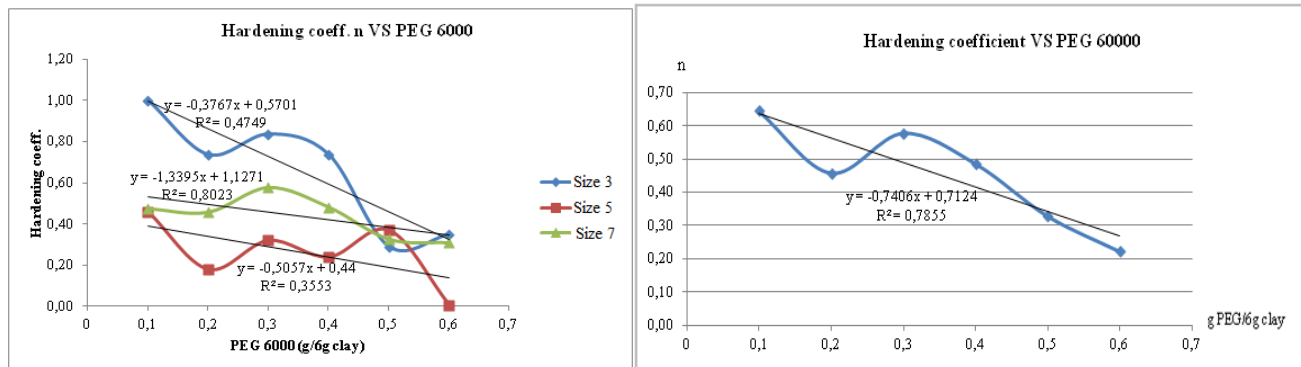
Figure 6 shows that the Young modulus increases significantly with the finenesses of grinding. This behavior is realistic since the finenesses of grinding permits a best agglomeration of clay material in general.

### 3.1.3. Hardening coefficient $n$

The figures 7-a) and b) show the evolution of the hardening coefficient “ $n$ ” according to the PEG 6000 and grinding size. We observe a great decrease of this coefficient according to PEG 6000.

According to figure 7-a), we can say that the material evaluate from a perfectly elastic material for the configuration (size 3, PEG 0.1 g) with a value of “ $n=1$ ” to a perfectly plastic material for the

configuration (size 7, PEG 0.6 g) having a value of “ $n=0.12$ ”. Figure 7-a) shows the regression line with a  $R^2$  up to 0.7. Thus, the plastification of the material follows a linear law according to the PEG 6000 addition.



**Fig. 7.** Hardening coefficient “n” according to a) grinding size and PEG 6000 b) PEG 6000

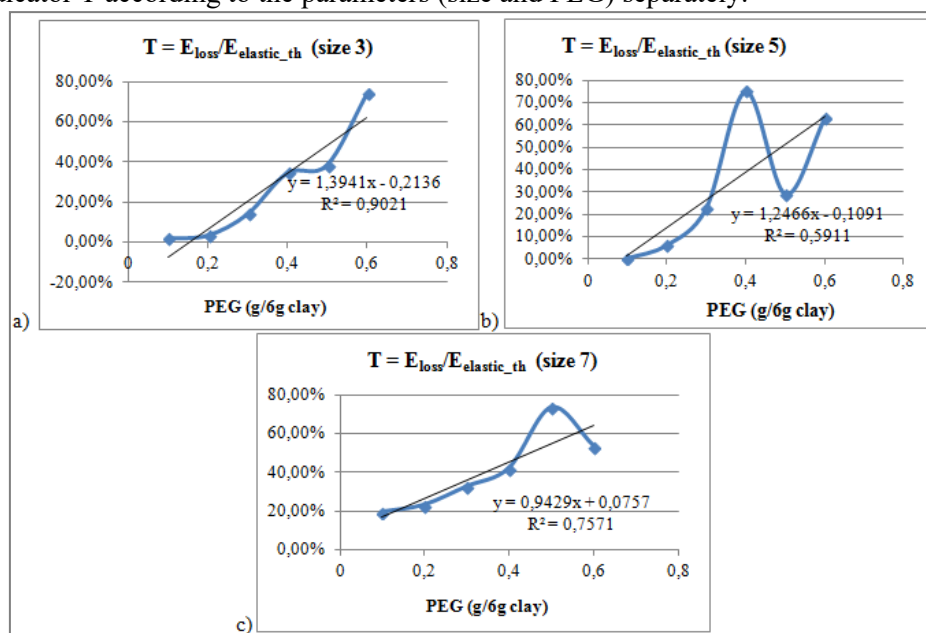
### 3.1.4. Energetic approach

The energetic analysis permits us to quantify the significant evolution of the materials according to the addition of the polymer. The approach is to evaluate the loss of energy caused by plastification phase. So for each material:

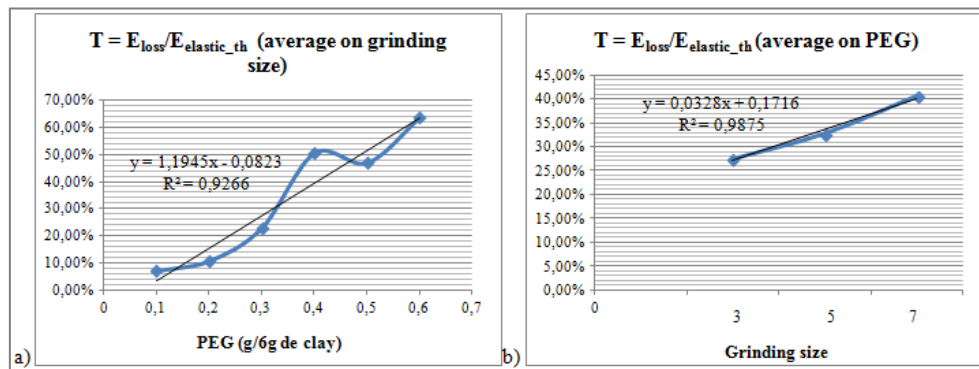
- According to the values of the Young modulus, we suppose a perfectly elastic material. We estimate the theoretical elastic energy that has to be created within sollicitation;
- We calculate in parallel the value of plastic energy transmitted within the material, according to the result of compression test
- The difference between the two energy quantities corresponds to the loss of energy caused by plastification.
- Thereafter, we estimate the loss of energy related to the theoretical elastic energy in term of percentage, as shown in equation 3 below :

$$\text{Relative loss of energy } T(\%) = \frac{\text{Loss}}{\text{theoretical Elastic energy}} \quad (3)$$

Figures 8-a), b) and c) show the evolution of the energetic ratio T according to the grinding size and PEG 6000 concentrations. We can observe a really significant and linear evolution of the relative loss of energy according to the PEG concentration. The figures 9-a) and b) shows the average of the energetic indicator T according to the parameters (size and PEG) separately.



**Fig. 8.** Relative loss of energy T according to grinding size and PEG 6000 doping



**Fig. 9.** Relative loss of energy  $T$  according to grinding size and PEG 6000 doping (averages)

From figure 9-b, we can see that the loss of energy increase also according to the grinding size. From our point of view, we interpret this result by the increasing of the inter-particle water that could exist within the clay matrix with the increasing of the finenesses of the grinding.

#### 4. Conclusion

The results presented in this work permit us to see that a simple procedure of a dry controlled grinding permits a significant evolution of plastification of the natural and not purified clay with the addition of the PEG 6000. Indeed the addition of the polymer does not affect the mechanical properties in the elastic domain, but in the plastic domain, the effect is very significant: the clay material passed from a perfectly elastic material to a quasi perfectly plastic material in the plastic domain.

A future step consists on the performing of more mechanical essays, related to vibration behavior of the new materials, to estimate the possibility of existing of a hysteresis related to charging/discharging cycles. If existing, the new materials or even the PEG polymer could be used in reinforcing ground against vibration and seismic solicitations.

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