

Effect of calcium solution on the mineralogy of the clay fraction and the swelling of clay soils

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Abstract

This article addresses the subject of treating clay soils to reduce the swelling phenomenon in this type of soil. A study was carried out by using a calcium-rich solution, and then we examined the influence of this latter on the clay fraction mineralogy (fraction $< 2 \mu\text{m}$), on the non-clay fraction, and on the clay soil swelling. This study is also interested in the dissolution of clay fraction minerals during this treatment. A mineralogical and free swelling analysis was carried out on three natural clay soils from three different locations in Algeria. The results reveal that the solution rich in ions (Ca) has a significant influence on the clay's mineralogy, since all the clay minerals have almost disappeared and only traces of smectite, illite, chlorite, and small quantities of kaolinite remain. The near disappearance of minerals with the calcium solution may be linked to the high pH of the pore water, because the increase in pH accelerates the minerals alteration. The pH of the solution induces significant modifications in the samples, revealing the high reactivity of the minerals during this treatment. Furthermore, it was observed that the treatment allows for a nearly complete reduction in the water sensitivity of these soils (i.e., swelling).

Keywords: Clay fraction, calcium solution, clay treatment, mineralogy, swell pressure

Clay and marly soils have a potential to exhibit undesirable geotechnical properties, such as swelling and high water sensitivity (Holtz & Gibbs 1956; Seed *et al.*, 1962; Diamond & Kinter, 1965; Eades & Grim, 1966; Sakr *et al.*, 2009). The main factor triggering the swelling phenomenon is the clay fraction nature. In particular, high smectite content with the presence of water causes soil swelling. Swelling soils exist in many regions in Algeria, particularly in the Batna region (N'Gaous, Boumagueur) and Oum El Bouaghi (Ain El Beida) and they create difficulties for the construction of buildings and the construction of major infrastructures, such as railways and motorways. Many studies show that treatment with lime and hydraulic binders improves the geotechnical properties of many soils (Ashraf and Walker 1963; Le Runigo *et al.*, 2009; Al-Mukhtar *et al.*, 2012; Zhao *et al.*, 2016; Etim *et al.*, 2017), making them suitable for construction applications of civil engineering works. Lime causes particle flocculation, which is mainly responsible for improving the engineering properties of soil (reducing its plasticity and swelling) and also leads to a pozzolanic reaction giving the cementitious compounds formation such as calcium silicate hydrates (CSH), calcium aluminate hydrates (CAH), and calcium aluminosilicate hydrates (CASH), which allows it to acquire a low plasticity and low swelling potential. The evolution of pozzolanic activity strongly depends on the mineralogy of the clay fraction (fraction $< 2 \mu\text{m}$) (Deneele *et al.*, 2016; Maubec *et al.*, 2017; Vitale *et al.*, 2016a; Vitale *et al.*, 2016b; Vitale *et al.*, 2017). The dissolution of clay minerals and the formation of pozzolanic materials give the main cementing agents CSH, CAH and CASH which are responsible for solidification and give high mechanical resistance to the treated soils (Bell, 1996). Clay minerals show different dissolution rates in alkaline environments, depending on layer charge, structure, and chemical composition (Elert *et al.*, 2018). Many studies

have focused on the reaction of clay minerals with lime (Bauer and Berger, 1998; Rozalen *et al.*, 2008). Maubec *et al.*, (2017) studied the effects of lime on two different clays, kaolin and bentonite. They showed that improvements are faster with Ca²⁺ bentonite; the performances achieved for kaolinite are higher, but in the long term. Vitale *et al.* (2017) have shown that flocculation due to cation exchange reactions controls the microstructural characteristics of kaolin and also the low reactivity of kaolinite-type clay minerals to pozzolanic reactions. Regarding lime-treated bentonite, they have noticed that the chemo-physical evolution depends on the combined effect of cation exchange reactions and pozzolanic reactions that develop on a comparable time scale.

Few studies were carried out on the solution rich in calcium's effect on the mineralogy of clays. We can mention (Vitale *et al.* 2016a), who studied the influence of pore water chemistry on the short-term behavior of kaolin treated with lime, and they showed that lime increased the average aggregate size of soil particles in the short term. (Chemeda *et al.*, 2018) looked at the interface chemistry of a lime solution and kaolinite in the short term and its effect on pozzolanic activity in the long term. They showed that the adsorption of calcium increases with the increase in its initial concentration, the higher the adsorbed calcium quantity, the lower the measured silicon and aluminum concentration. (Vitale *et al.*, 2017) and (Chemeda *et al.*, 2015) show that the low reactivity of kaolinite clay is compatible with the consumption late of portlandite and the formation of new hydrated phases. Chemeda *et al.*, (2015) proved that Ca ions at higher pH (CaOH⁺) mainly favor the face–face bonds between kaolinite particles, which are effective in forming tight flocs with lower deformation. The flocs behave like individual coarse-grained materials. Thus, their reactivity with water should be reduced during exposure to humidity, which in turn reduces the swelling-shrinkage potential and improves their workability. On the other hand, Bauer and Berger (1998) demonstrate that in an alkaline solution, the dissolution of kaolinite is greater than that of montmorillonite. According to Maubec *et al.* (2017) this contradiction can be explained by another mechanism that occurs in parallel with the kaolinite dissolution.

The stabilization and improvement of clay soils has been widely studied by several researchers (Diamond and Kinter, 1965; Eades and Grim, 1966; Bell, 1996, Hatim and Mostafa 2017, Amri *et al.*, 2019). However, the chemo-mineralogical mechanism, in particular the effect of the treatment on clay minerals (smectite, illite, kaolinite, muscovite, etc.), is not yet fully understood. In this experimental work, we study the influence of a solution rich in calcium on the mineralogy of the clay and non-clay fraction as well as on the swelling of swelling marly soils of Miocene age, which outcrop in the N'Gaous, Boumagueur and Ain el Beida regions. This marly soil layer poses engineering problems for the construction of civil engineering works. We will study the chemo-mineralogical evolution of the treated samples, and we will focus on the relationships between the mineralogical evolution and the macroscopic behavior (swelling) of the studied soils.

Materials and methods

Soils used and admixtures

The soil used in this study is green marl of Miocene age taken from three different localities in Algeria, in particular in the regions of N'gaous, Boumagueur (Wilaya of Batna), and Zorg (Wilaya of Oum El- Bouaghi). The region of N'gaous is a commune of the Wilaya of Batna (Algeria), located 92 km southwest of the capital of the Wilaya of Batna. Boumagueur is located 5 km south of the daïra of N'Gaous and 85 km west of the wilaya of Batna. While the commune of Zorg is part of the high plains of Constantine, it is located 47 km northeast of the Wilaya of Oum El

Bouaghi (Fig. 1).The presence of clay formations (marl of Miocene age) makes these areas sensitive to swelling phenomena and creates difficulties in constructing civil engineering works.

Sample preparation

The soil has been dried in an oven at a temperature of 50°C and then has been ground into a powder. After that, it has been sieved through a 2 mm sieve. Marl soil and the admixture (calcium solution) have been mixed and homogenized for 10 min. The percentages of the admixture used in the mixtures are 0%, 2%, 3%, 5%, 7% and 9% for the three samples. Therefore, the samples have been mixed with distilled water. The mixture has been statically compacted in a CBR press at a slow speed (2 mm/min) in rigid steel rings (70 mm diameter and 19 mm height). The samples have been immediately wrapped using both cellophane and aluminum paper, and then they have been coated with paraffin wax to avoid the loss of humidity before being stored at 20° C for 28 days.

In the case of treated soils, a quantity of the admixture corresponding to the dosage in question was added to the soil. This quantity is calculated using the following expression:

$$\text{Admixture \%} = \frac{\text{masseofadmixture}}{\text{masseofadmixture} + \text{masseofdrysoil}}$$

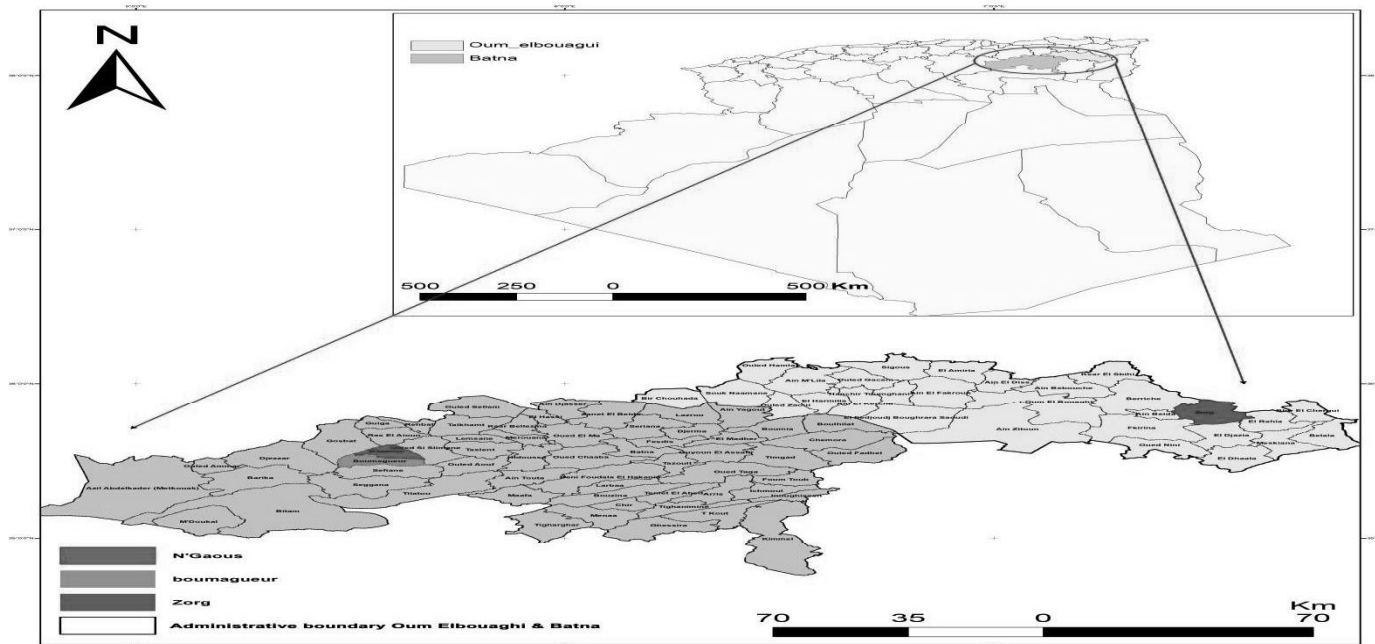


Figure 1. Geographic location of the sampled soil area.

Geotechnical analysis

Physical test

A set of laboratory tests has been conducted with the aim of defining the physical properties of the soil. These tests are:

Volumetric mass (apparent density): The samples are subjected to the water immersion method to determine their volumetric mass, in accordance with French standard NF P 94-053. The density ρ is the result of dividing the mass (m) of the sample by its volume (V). In order to be paraffin, the sample to be tested must have a simple shape. The test sample, after any size, is immediately weighed (m) and is waxed immediately afterwards. It is weighed (mp) in the open air, then placed in the basket suspended from the balance bracket and immersed in order to be weighed (mp') with the hydrostatic

balance. The three weighings make it possible to determine the density by the relations:

$$\rho = \frac{m}{V}$$

With: $V = (m_p - m'_p) \times \rho_p / \rho_w - (m_p - m)$

ρ_w : volumetric mass of water

ρ_p : volumetric mass of paraffin.

The granulometric analysis aims to define the distribution of the constituent grains of a soil by granulometric class. This analysis was determined according to the NF P 94-056 and NF P 94-057 standards, it is carried out by sieving for grains greater than 80 μm and by a sedimentation test for particles less than 80 μm . Particles of 80 μm are dispersed in sodium hexametaphosphate, then stirred in order to homogenize the sample. The sedimentation rate of the particles varies according to their size, and based on Stokes' law, to establish the distribution of the particles according to their sedimentation time. The dimensional distribution curve of the soil elements often allows its geological identification. In addition, it always offers an interesting first approach to its geotechnical qualities, in particular by knowing the relative importance of the clay phase.

The Atterberg limits were determined according to the NF P 94 051 standard. The plasticity index is commonly used to measure clayiness. This can be presented in different states of consistency, which are a function, on the one hand, of the quantity of interstitial water and, on the other hand, of the quantity of adsorbed water. A Swedish agricultural engineer, Atterberg, and then subsequently Casagrande (1947), conventionally defined, from the water content of the medium, the limits of various states of consistency of a given soil:

The liquid limit (WL): relatively high water content at which the soil passes from the plastic state to the liquid state. Plastic limit (WP): relatively low water content at which the soil passes from the solid state to the plastic state. The difference in water content between the liquid and plastic limits represents the plasticity index of the soil.

The methylene blue test (VBS) is carried out according to the NF P 94-068 standard. The methylene blue value is another measure of clayiness, this parameter represents the quantity of methylene blue necessary to saturate a suspension of water, or in other words, the quantity of blue that can be adsorbed on soil particles. Since it is the clay particles that have the greatest capacity to adsorb methylene blue, this test therefore provides an overall estimate of the quantity and activity of the clays contained in the soil. The VBS is expressed in g of blue per kilo of dry soil.

Free swelling and expansion pressure test

Swell pressure is determined using the free swelling method. After assembling the sample in the Oedometer cell, the latter is forwarded to the imbibition process under a stress corresponding to the weight of the piston, varying between 0.7 and 6.3 kPa. The vertical deformations are measured. The maximum deformation related to the initial height corresponds to the swell potential. Once the deformation of the expansion is stabilized, the loading is carried out gradually, step by step, according to a chosen loading rate. The swell pressure corresponds to the load that has to be applied in order to get the sample back into its initial height.

mineralogical and microstructural analysis

The mineralogical composition of the total sediment (disoriented powder) and the composition of the clay fraction ($< 2 \mu\text{m}$)

were analyzed by X-ray diffraction using a Bruker D8 Advance diffractometer (Department of Geology, AGE, University of Liège, Belgium). Operating at 40 kV and 25 mA using Cu-K α 1 radiation ($\lambda = 1.5406 \text{ \AA}$). To prepare and analyze the total disoriented powder, the sample was dried in an oven at 40 °C, manually ground in a mortar, and then sieved at 250 μm and placed on a PVC sample holder, then lightly compacted from behind, and the supports are closed so as not to influence the preferential orientation of the mineral particles contained. They are finally subjected to X-ray radiation scanning at an angle of 2° to $45^\circ 2\theta$ for the determination of the total mineralogical composition.

The clay slides (oriented sample) are prepared by direct deposition of a diluted suspension of particles smaller than 2 μm on a ground glass slide, followed by drying in the open air. The clay suspension prepared for X-ray diffraction was previously decalcified, and the clay fraction (less than 2 μm) was extracted after a decantation process based on gravitational sedimentation according to Stokes law. The first centimeter of the suspension is applied to a glass slide and dried overnight at room temperature (Moore and Reynolds, 1989). The type of clay minerals is determined from the analysis of 3 slides that have undergone different preparation protocols: open air drying (natural state), solvated with ethylene glycol (EG) for 24 h (highlighting of swelling clays), and heating at 500 °C for 4 h (H) (destruction of kaolinite). Qualitative identification was carried out by referring to the intermediate space (Brown and Brindley, 1980) and to the intensity values of the main reflections between the three X-ray diagrams, characteristic of each mineral. Semi-quantitative estimates are based on the measurement of the intensity of characteristic peaks measured on X-ray patterns. The semi-quantitative estimate was estimated using the correction factors of (Cook *et al.*, 1975; Boski *et al.*, 1998) for total disoriented powder and those of (Fagel *et al.*, 2007) for clay minerals.

Differential thermal and thermogravimetric analysis (DTA-TG) was performed on the samples using a DTA-TG device, TGA 2000 brand (Department of Geology, AGE, University of Liège, Belgium).

The surface state modifications and fabric changes of soil particles have been examined through scanning electron microscopy by using a JEOL JSM-U3 type apparatus. The operation of the scanning electron microscope is based on the interaction between an electron beam and a crystalline matrix or not. The images were obtained by a secondary electron detector. This technique provides information on the relief of the sample; the morphology of the grain and its disposition for untreated samples, but also information on the formation of cement materials for samples treated.

Table1. Mineralogical and geotechnical characteristics of the three samples

Measured parameters	N1	Boum2	OMB3
Clay minerals %	65	60	53
	26 % Smectite 28 % Kaolinite 15 % Illite	63 % Smectite 25 % Kaolinite 12 % Illite	64 % Smectite 23 % Kaolinite 13 % Illite
	5 % Smectite-Illite 26 % Chlorite	/	/
Non-clay minerals%	35	40	47
	16 % Quartz 13 % Calcite 6 % Gypsum	18 % Quartz 20 % Calcite 2 % Gypsum 0.5 % Feldspar 0.5 % Dolomite	9 % Quartz 34 % Calcite 1 % Gypsum 1.5 % Feldspar 1.5 % Dolomite
Clay fraction ($d < 2\mu$) % (NF P 94-056 et NF P 94-057)	64	54	50
Natural water content Wn (%) (NF P 94-050)	22.09	20.18	16.54
Wet density γ_h (t/m ³) (NF P 94-053)	2.02	2.06	2.17

Dry density γ_d (t/m ³) (NF P 94-053)	1.65	1.71	1.86
Degree of saturation S_r (%)	95.64	94.06	98.94
Liquid limit WL (%)	75	59	61
Plastic Limit Wp (%)	33	23	21
Plasticity Index Ip (%)	42	45	40
Swelling Coefficient Cg (%)	6,60	8,08	9,26
Swelling pressure Pg (kPa)	258	171	161
Methylene blue Value VBS (%)	8.6	7.8	7

Results and discussion

Mineralogical, physicochemical and geotechnical characteristics of natural samples

The characterization through mineralogical, physico-chemical and geotechnical analyses of the three taken samples, samples of the N'gaous area (N1), Boumagueur (Boum2) and Oum El-Bouaghi (OMB3), was studied.

The mineralogy of clay is essential in soil treatments, as clay minerals react differently with the treatment. The mineralogical composition was determined from X-ray diffraction spectra taken from a representative sample of the material and from clay slides of the fraction less than 2 μm . The first method, called powder diffractogram, makes it possible to identify all the minerals present (quartz, feldspars, calcite, gypsum, etc.) as well as phyllosilicates.

The presence of quartz, feldspars, calcite, dolomite, and gypsum is identified from the principal reflections at 3.33 Å, 3.20 Å, 3.03 Å, 2.89 Å, and 7.5 Å, respectively. The total fraction comprising phyllosilicates is detected by the presence of lines around 4.6 Å.

the slides of the clay fraction (fraction $<2\mu$) which are prepared by direct deposition of a dilute suspension of particles smaller than 2 μm . The type of clay minerals is determined from the analysis of 3 slides having undergone different preparation protocols: open-air drying (natural state), glycolation (demonstration of swelling clays), and heating (destruction of kaolinite). Thus, illite is detected by the presence of lines at 10 Å, 5 Å and 0.34 Å which correspond to the reflections (001), (002) and (003) of a phase unaffected by the treatment with ethylene glycol and heating at 550°C. Kaolinite is highlighted by the presence of (001) and (002) reflections at 7.15 Å and 3.5 Å in the natural state, not modified by ethylene glycol treatment, but which disappear after heating to 550°C, (dehydroxylation of kaolinite). The smectitic component is diagnosed by the presence, in the glycol diffractogram, of a (001) reflection developed at around 17 Å, which is established around 10 Å after heating to 550°C, by the departure of interfoliar water. Chlorite is detected by its reflection at 14 Å in the natural and/or glycol diffractogram. The intensity of the 14 Å reflection can increase or decrease upon heating, depending on its degree of degradation.

The results of the mineralogical study (Table 1) show that the three samples have a very similar mineralogical composition, predominantly consisting of smectite and kaolinite, with a smaller amount of illite.

Figure 2 illustrates the XRD patterns of the $b_{2\mu}$ fraction of the untreated sample (N1). The 7.1 Å reflection disappears after heating to 500 °C, confirming the presence of kaolinite. The 10 Å peak, stable regardless of the treatment (N, EG, H), is attributed to illite. The 001 reflection corresponding to 14 Å is attributed to chlorite and remains stable after treatments with ethylene glycol and at 500 °C. The presence of a reflection at 12,8 Å is attributed to an interstratified (smectite-illite) and a reflection 16 Å attributed to smectite.

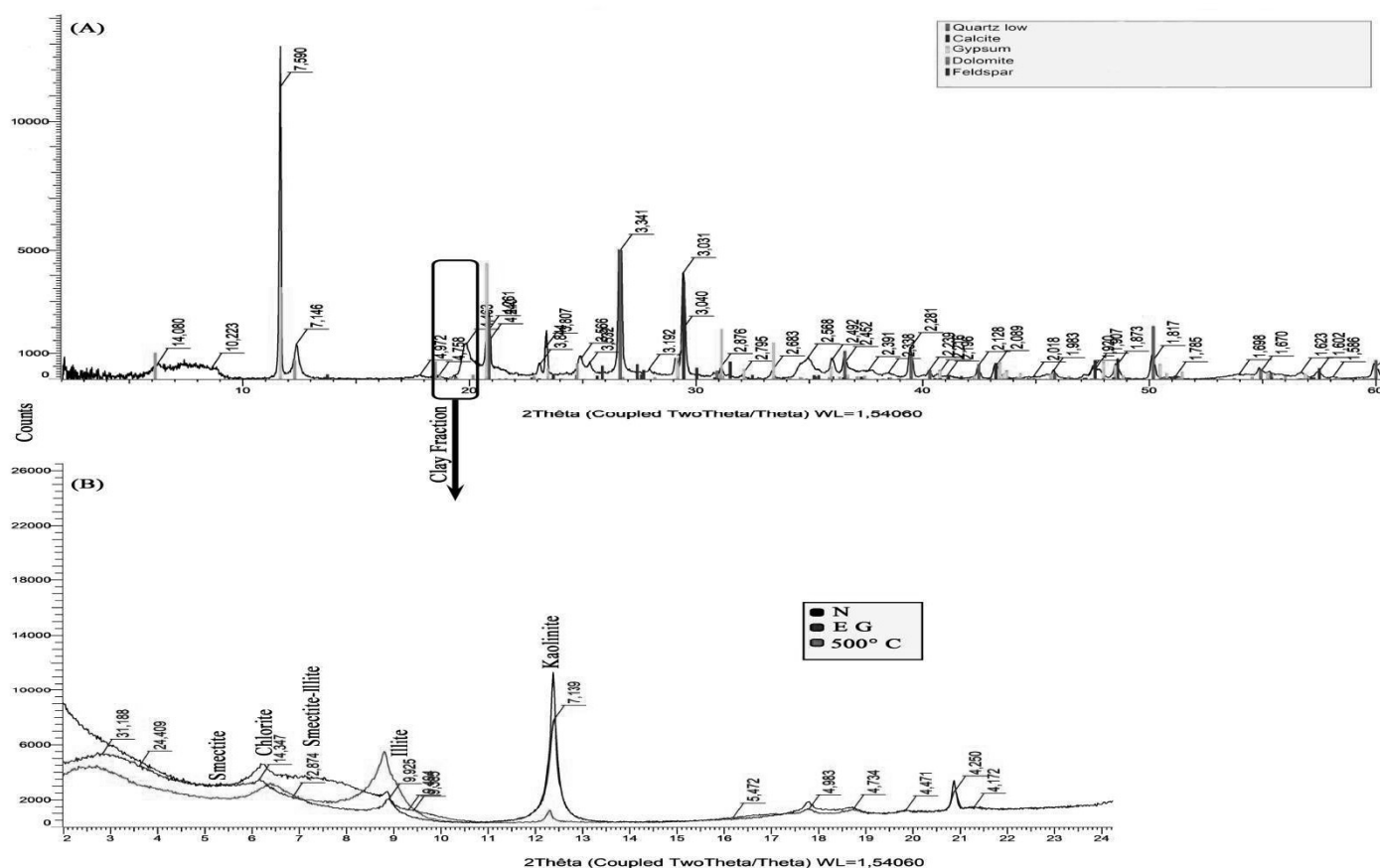


Figure 2. X-ray diffraction patterns of N1 sample ((A) total clay of sample; (B) clay fraction treated: (N) untreated sample; (EG) treated with ethylene glycol; heated at 500 °C).

Table 2 illustrates the chemical composition of samples, which was determined by X-ray fluorescence spectroscopy (XRF) with an apparatus type Panalytical PW4600 (Axios fast). The loss on ignition (LOI) was obtained by the gravimetric method at 1000 °C for 2 h. The results of the chemical analysis of the three samples confirm the results of the XRD mineralogical analysis. Sample N1 contains 48% silica (SiO₂), 17% alumina (Al₂O₃) and 7% carbonate (CaO), sample Boum2 contains 45% silica (SiO₂), 11% alumina (Al₂O₃) and 13% carbonate (CaO) while the OMB3 sample contains 30% silica, 9% alumina and 23% carbonate.

The high level of silica and alumina and the low proportion of carbonate in the two samples N1 and Boum2 indicate the high proportion of aluminosilicates in the two samples compared to the OMB3 sample which has a lower level of silica and alumina with a high proportion of carbonate.

The results of the physical and geotechnical analysis show that the three studied samples have a fine grain size (Fig. 3), they are very plastic and they are classified as very expansive soils (Table 1 & Fig. 4).

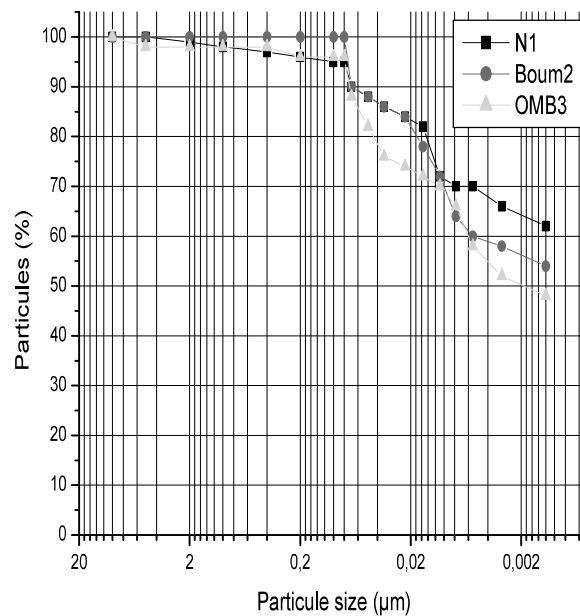


Figure 3. Particle size distribution of the samples studied

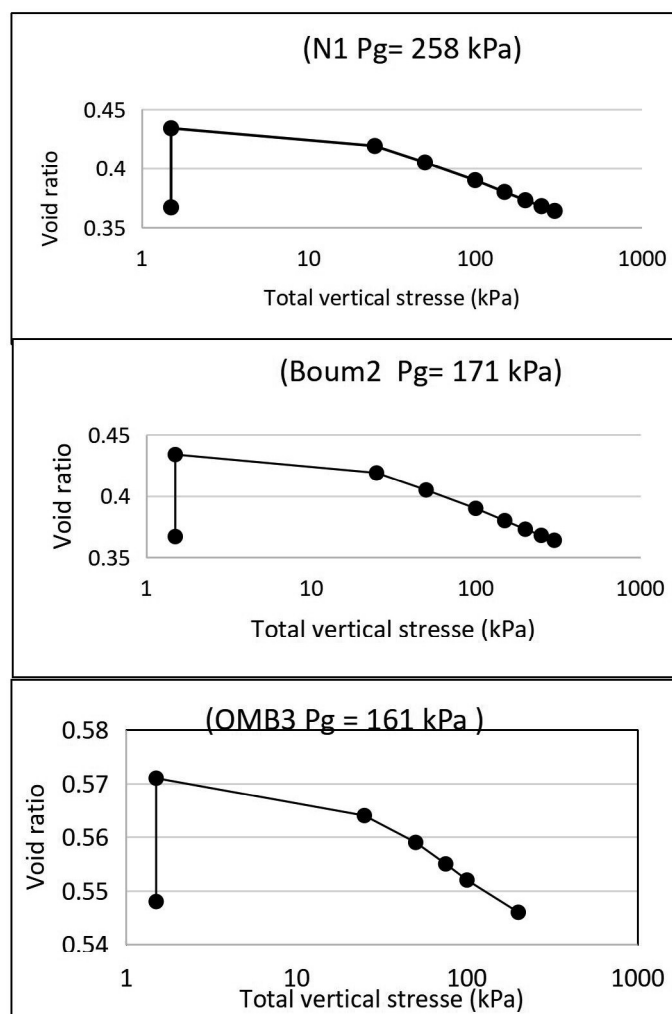


Figure 4. Swelling pressure of the samples studied

Table 2. X-ray fluorescence results on pearl (integration of law) in%.

Sample	SiO2	TiO2	Al2O3	Fe2O3	MnO	MgO	CaO	Na2O	K2O	P2O5	LOI	SOMME
N 1	48,02	0,79	17,02	6,56	0,37	2,57	7,25	0,52	2,06	0,22	14,56	99,93
Boum2	45,23	0,60	11,83	4,75	0,03	2,50	13,18	0,45	1,61	0,24	18,47	98,89
OMB3	30,18	0,52	9,66	4,02	0,03	1,52	23,99	0,23	0,74	0,08	27,93	98,89

The used adjutant in this study is a saturated solution of calcium hydroxide ($\text{Ca}(\text{OH})_2$), produced by mixing slaked lime (Fig. 5) with water (CRR, 2010). Table 3 summarizes the characteristics of this product.

Table 3: Components of the adjutant (CRR, 2010)

Components	Quantity
$\text{Ca}(\text{OH})_2$	400 g
H_2O	1 litre

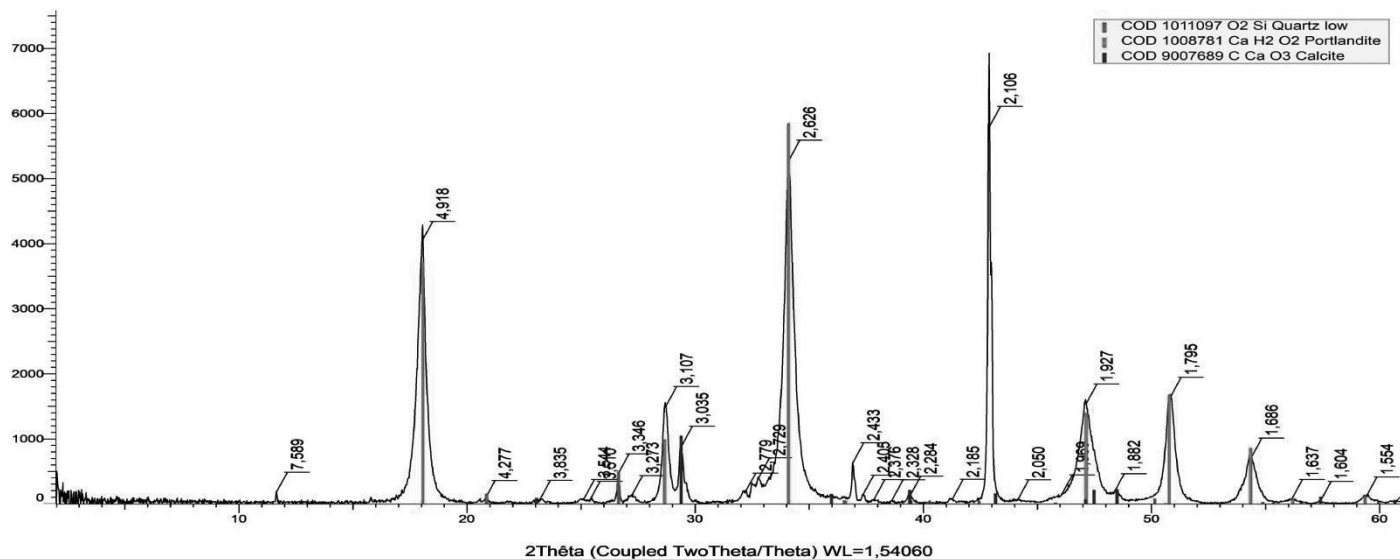


Figure 5. X-ray diffraction patterns of the used slaked lime

Effect of the calcium solution on the swelling of samples

The swelling pressure evolution of the untreated and treated samples and cured for 28 days are collated in Table 4. five solution contents were used in this study: 2%, 3%, 5%, 7% and 9%. The treated sample will be compared to an untreated sample considered as a reference. The test specimens prepared for swelling pressure measurements were fabricated to have conditions closely resembling their natural state.

Table 4. solution additions influence on the swelling of the samples.

Admixture %	N1 Pg (kPa)	Boum2 Pg (kPa)	OMB3 Pg (kPa)
0	258	171	161
2	127	117	47
3	60	109	34
5	36	95	11
7	21	72	9
9	8	46	

The table results show that the sample N1 in his natural state is classified as a highly swelling soil with a swelling pressure of 258 kPa. As shown in Fig. 6, The swelling reductions obtained were proportional to the amount of calcium solution added. When the solution dosage is low (2% and 3%), the swelling pressure decreases from 258 kPa to 127 kPa and 60 kPa. For higher doses, 5%, 7% and 9%, a significant reduction in swelling pressure was observed, and the samples showed values of swelling pressure of 36, 21 and 08 kPa, respectively. We therefore note that an increase in the calcium solution content significantly reduces the swelling pressure of the studied sample.

The treatment of the Boum2 sample with the same addition gives results close to that of the N1 sample. The results show that adding 2%, 3%, 5%, 7% and 9% calcium solution to the sample gave swelling pressures of 117, 109, 95, 72 and 46 kPa, respectively.

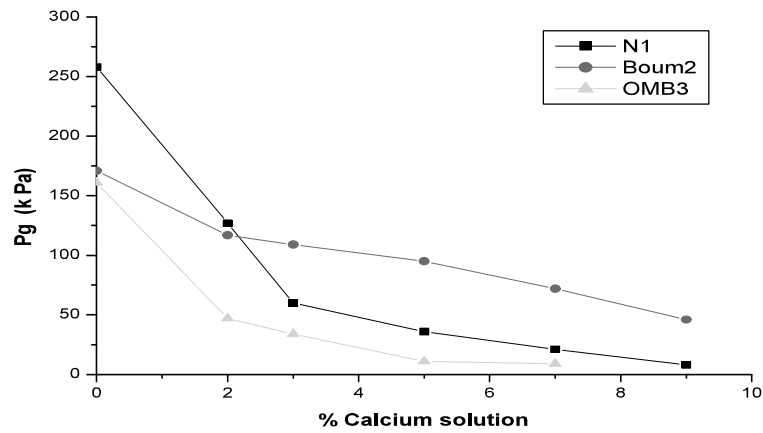


Figure 6. The influence of calcium solution addition on the swelling pressure of the three studied samples.

Concerning the OMB3 sample, the influence of the calcium solution seems more important than the other two samples. From the results we see that the swelling pressure decreases significantly with a small percentage of the added calcium solution (2% to 3%); beyond this percentage, the swelling becomes negligible and the pressure swelling stabilises.

The stabilisation method by adding a calcium solution seems very effective in reducing swelling pressure, because with low percentages of addition, there is a significant reduction in swelling pressure. This reduction in swelling pressure is significant for the three tested samples, and the best reductions are obtained by adding a small percentage (2% to 3%) of calcium solution.

Effect of calcium solution on the mineralogical and microstructural properties of samples

The marl formations of the three samples (N1, Boum2 and OMB3), untreated and treated at different percentages of the calcium solution, are characterised mineralogically in order to evaluate the reactivity of the minerals (clay and non-clay) with this solution and the formation of cementitious phases.

The evolution of chemo-mineralogical and microstructural properties during treatment was studied using XRD, DTA/TG analyses and scanning electron microscope (SEM). X-ray diffraction is used to highlight crystallised phases such as portlandite and C-A-H, C-A-S-H and C-S-H type phases.

279 **Table 5. Mineralogy of treated and untreated samples.**

Sample	Mineralogy of the raw sample %										Clay mineralogy (fraction <2μ) %									
	Gypsum (d=7,83 - 7,50)		Calcite (d=3,04- 3,01)		Quartz (d=3,37– 3,31)		Clay fraction (d=4,46)		CSH (d=3,04)		Smectite (d=17)		Kaolinite (d=7,25- 7,02)		Illite (d=10,00)		Smectite-Illite (d=14,00 - 10,00)		Chlorite (d=14,00)	
	Corrective factors of each ineral										Corrective factors									
	(0,40)		(1,65)		(1,00)		(20,00)				(1,50)		(0,70)		(1,00)		(0,40)		(0,34)	
	Cook et al., (1975)		Cook et al., (1975)		Cook et al., (1975)		Boski et al., (1998)				Fagelet al., (2007)		Fagelet al., (2007)		Fagelet al., (2007)		Fagelet al., (2007)		Fagelet al., (2007)	
	Intensity	relative abundance%	Intensity	relative abundance%	Intensity	relative abundance%	Intensity	relative abundance%	Intensity	relative abundance%	Intensity	relative abundance%	Intensity	relative abundance%	Intensity	relative abundance%	Intensity	relative abundance%	Intensity	relative abundance%
N 1 (untreated sample) :					% no clay minerals :35										% clay minerals : 65					
0 % Calcium solution	3,5	6	2	13	4,5	16	1,1	65	0	0	0,2	26	3,2	28	0,6	15	0,3	5	0,6	26
3 % Calcium solution	3,5	6	2	13	4,5	16	1	-	0,8	+	0,1	13	3	26	0,5	13	0,3	5	0,5	22
5 % Calcium solution	3,5	6	2	13	4,5	16	1	-	1,5	++	0	0	2,2	19	0,2	5	0	0	0,1	4
B 2 (untreated sample) :					% no clay minerals : 40										% clay minerals : 60					
0 % Calcium solution	1,1	2	5	20	6,5	18	1,1	60	0	0	4,2	63	4,1	25	1,2	12	/	/	/	/
3 % Calcium solution	1,1	2	5	20	6,5	18	1	-	0,3	+	2,5	38	3,3	20	0,8	8	/	/	/	/
5 % Calcium solution	1,1	2	5	20	6,5		18	--	1,2	++	2,3	35	3,3	20	0,7	7	/	/	/	/
OMB 3 (untreated sample) :					% no clay minerals : 47										%clay minerals : 53 %					
0 % Calcium solution	1	1	3,7	34	7,3	9	0,6	53	0	0	1,2	64	2,2	23	0,5	13	/	/	/	/
3 % Calcium solution	1	1	3,7	34	7,3	9	0,6	/	0,2	+	6,2	/	6	/	0,5	/	/	/	/	/
5 % Calcium solution	1	1	3,7	34	7,3	9	0,4	--	0,3	++	0,1	4	1,6	17	0,1	3	/	/	/	/

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281 *Effect of treatment on non-clay fraction (total clay)*

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283 X-ray diffraction analysis was performed on the three treated and untreated samples to confirm the formation of new

284 minerals that may play a significant role in the swelling behavior of stabilised soils.

285 The intensities reflect the relative abundance of different clay and non-clay minerals in the material. The relative

286 abundance of minerals was estimated from measuring the characteristic height of the main peak reflections for each

287 identified mineral, multiplied by a specific correction factor. Fig. 4 shows X-ray diffractograms of sample N1 in its

288 natural state and treated after 28 days. The sample in its natural state is essentially composed of 65% clay, 16% quartz,

289 13% calcite and 6% gypsum. To evaluate the effect of the calcium solution on this sample, we present in the same Fig.

290 7 the results obtained after treatment and the main minerals existing in the samples treated with 3% and 5% calcium

291 solutions.

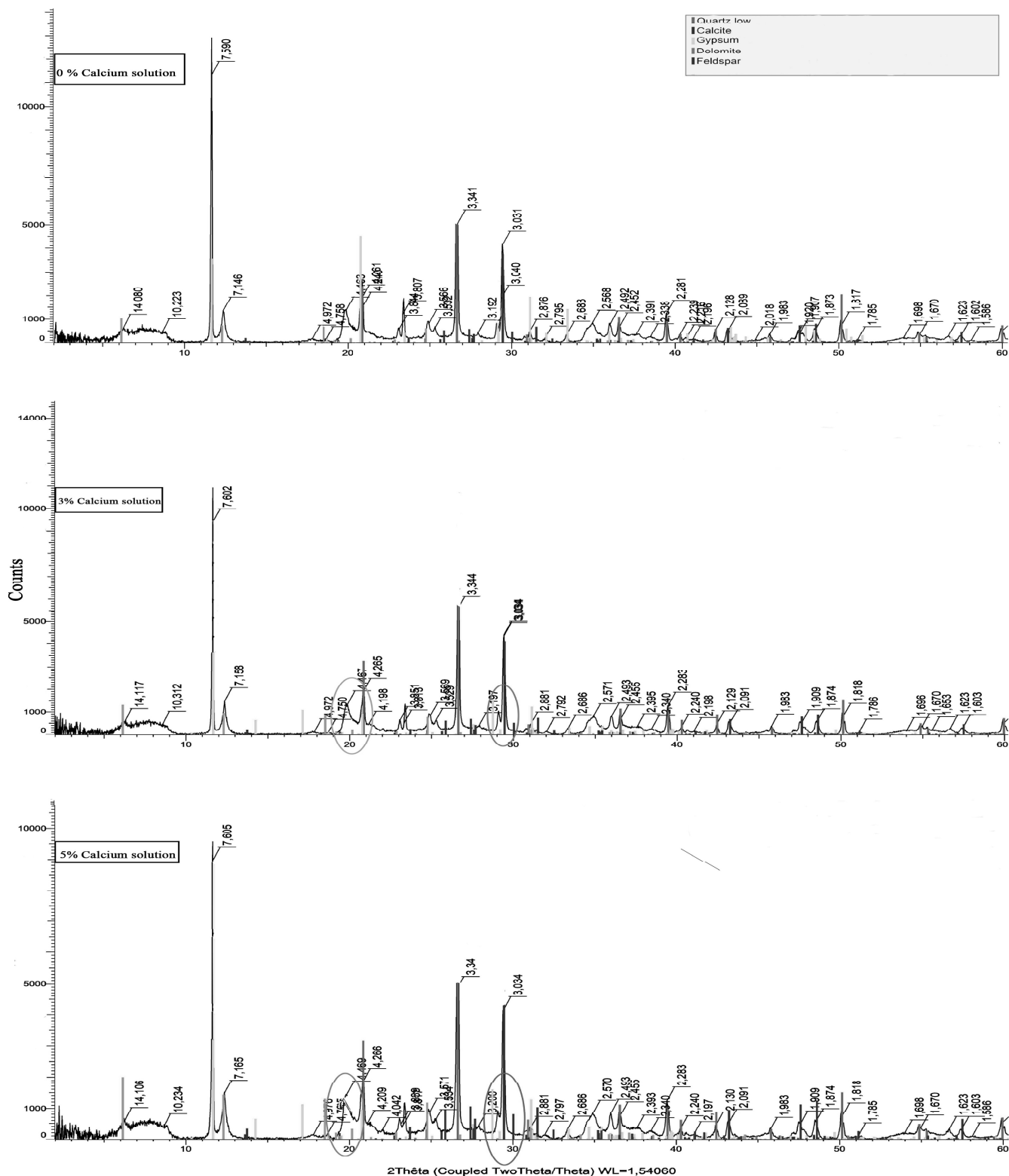


Figure 7. X-ray diffraction of the total clay of the sample (N1) untreated and treated with calcium solution. The peak corresponding to CSH. Clay fraction.

The comparison of the three diffractograms shows the appearance of new peaks indicating the formation of pozzolanic products (CSH) in the treated samples. The small peak corresponding to CSH can be observed in the treated soil spectra next to the calcite peak (in the range of 2θ $29.0^\circ - 30.0^\circ$). The clay fraction peak (phyllosilicates) is generally identified by its common reflection at $4.46 \text{ \AA}$. The absence of portlandite ($\text{Ca}(\text{OH})_2$) at $2\theta = 18.00^\circ$ in the samples

301 treated with 3% and 5% of calcium solution addition confirms the total consumption of this calcium-rich solution by
302 the reactions.

303 The results show that there are variations in intensity and relative abundance of the clay fraction ($d = 4.46 \text{ \AA}$) during
304 the treatment which decrease with the increase in the percentage of addition, which shows that there is reactivity of this
305 fraction during treatment. Alongside this reduction, the reflection intensity of cementitious compounds (CSH) appears.
306 Regarding the intensity and relative abundance of the other non-clay minerals (16% quartz and 6% gypsum), we note
307 that they were not reduced during treatment; this indicates that these two minerals were not influenced by the treatment
308 (Table 5). Other cementitious compounds (CAH and CASH) were not observed in the treated samples.

309 For the Boum 2 sample (Fig.8 & Table 5), which is essentially made of 60% clay, 18% quartz, 20% calcite, 2%
310 gypsum and traces of feldspars and dolomite. The results show that the sample was subjected to mineralogical change
311 and the formation of new minerals after solution treatment. The treated samples show the formation of CSH type
312 cementitious compounds. However, the peak intensity linked to cementitious compounds appears clearer with 5%
313 calcium solution contents; conversely, the other cementitious compounds (CAH and CASH) were not detected in the
314 treated samples.

315 The peak intensity of the clay fraction decreases with the increase in the calcium solution percentage. The intensity and
316 relative abundance of other non-clay minerals were not reduced during treatment, indicating that these minerals were
317 not influenced by treatment.

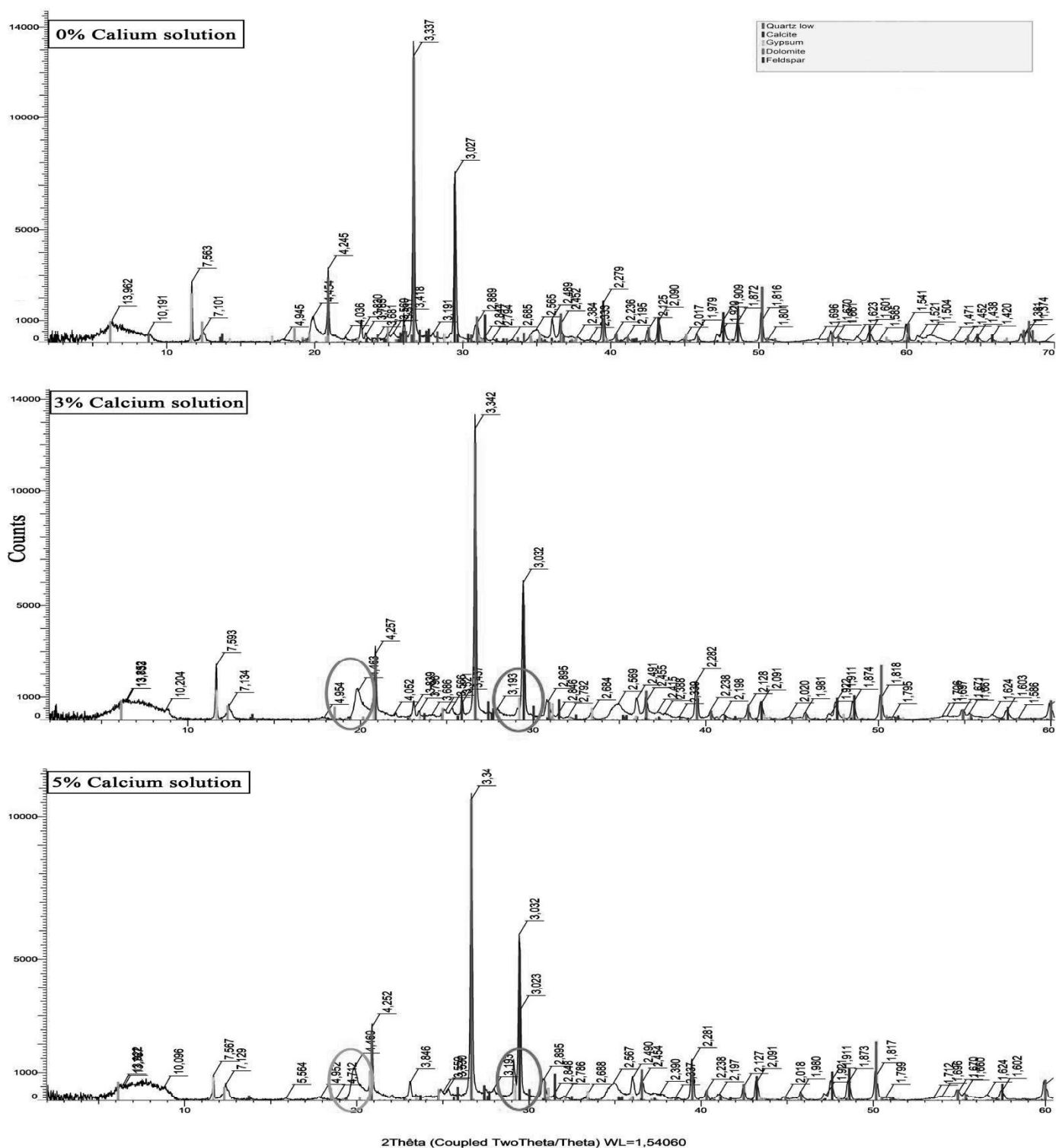
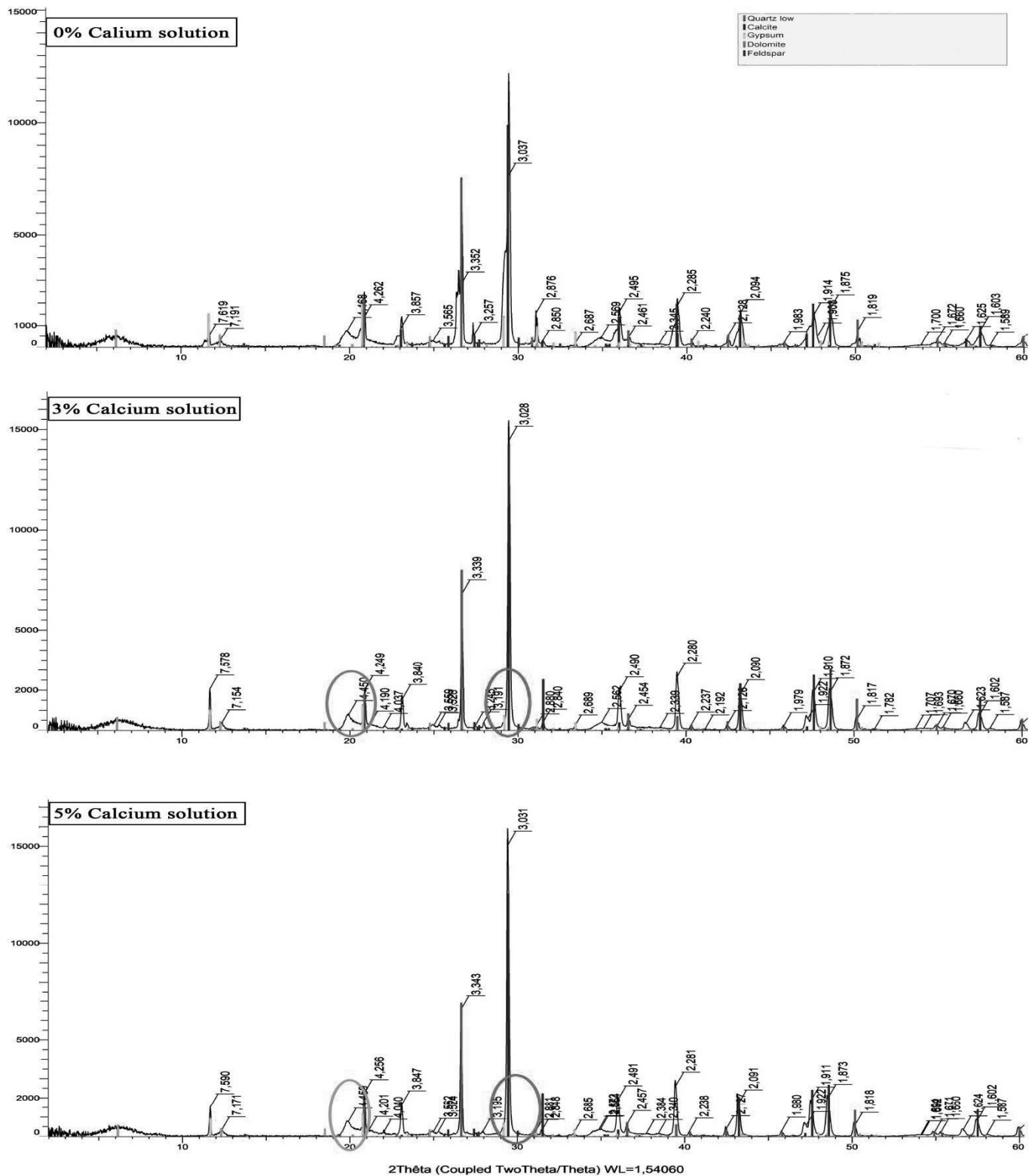


Figure 8. X-ray diffraction of the total clay of the sample (Boum2) untreated and treated with calcium solution. The peak corresponding to CSH Clay fraction

In the case of sample OMB3, results similar to the two previous samples were obtained. Indeed, as shown in Fig. 9 and Table 5, the presence of a low intensity of the peaks of the CSH compounds for the samples treated at 5%. The peak intensity of the clay fraction decreases with the increase in the calcium solution content; the decrease in the reflection of the clay results from the reaction of this fraction with the stabilizing additions, and the main mineralogical modification was observed during treatments with 5% of calcium solution content. The absence of a portlandite peak in the diffractograms of the treated samples proves the complete consumption of the added calcium.



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Figure 9. X-ray diffraction of the total clay of the sample (OMB3) untreated and treated with calcium solution. The peak corresponding to CSH ○ Clay fraction ○

Effect of treatment on the clay fraction (fraction < 2 μm)

The X-ray diffraction (XRD) patterns of an orientated slide of the untreated and treated samples with 3% and 5% from the three samples N1, Boum2 and OMB3, at a curing time of 28 days, are shown in Fig. 10, 11, 12 and Table 5.

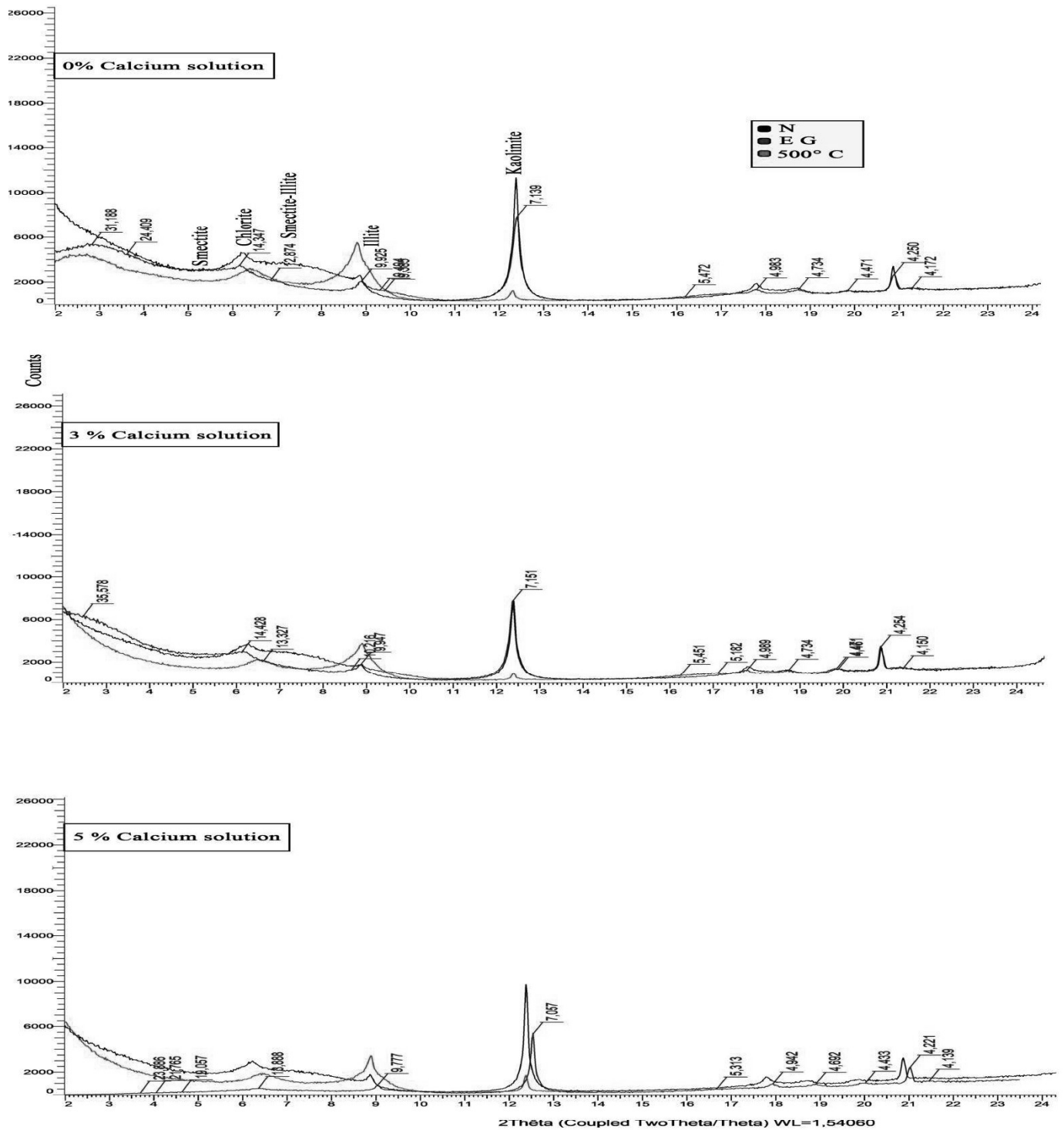


Figure 10. X-ray diffraction of an oriented slide of the sample (N1) untreated and treated with calcium solution.

The intensities reflect the relative abundance of different clay minerals in the material. The relative abundance of minerals was estimated from measuring the characteristic height of the main peak reflections for each identified mineral, multiplied by a specific correction factor.

Recall that the clay fraction of the natural sample N1 (untreated) is essentially composed of 26% smectite, 28% kaolinite, 15% illite, 5% smectite+illite, and 26% chlorite. Fig. 10 and Table 5 shows the variation in the relative abundance of clay minerals for the untreated samples and those treated with different calcium solution contents (3%

and 5%). The figure shows that stabilizing the sample with a calcium-rich solution results in a reduction in the intensities of all clay minerals. The clay mineral peaks decrease with increasing addition percentage. At 3% calcium solution addition, the reduction in smectite intensity, which decreased from 26% to 13%, illite from 15% to 13%, and chlorite from 26% to 22% are significant, and all these minerals have virtually disappeared at a 5% addition percentage. The kaolinite peak changes little, which indicates a low reactivity of this phase in the material. This study shows that the calcium solution has a significant effect on the mineralogy of this marl sample.

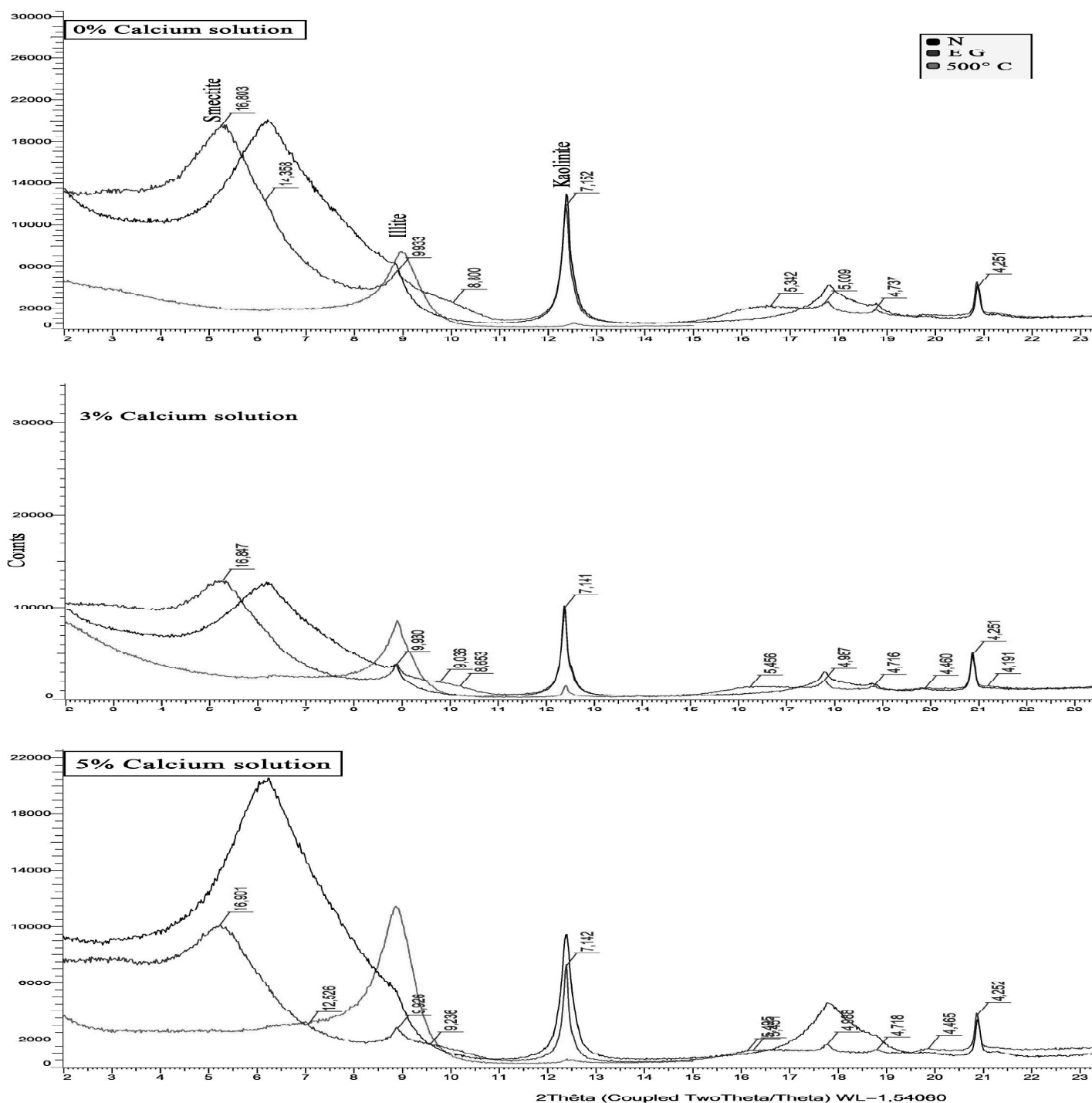


Figure 11. X-ray diffraction of an oriented slide of the sample (Boum2) untreated and treated with calcium solution.

The variation in the intensity of clay minerals in sample Boum2 as a function of the percentage of calcium solution addition is shown in Fig. 11 and Table 5. The diagrams of the Boum2 samples treated with 3% and 5% calcium solution after a maturation time of 28 days were compared with those of the untreated sample, composed mainly of 63% smectite, 12% illite and 12% kaolinite. The results show variations in the intensity and relative abundance of the minerals in the clay fraction during treatment, which decrease with increasing percentage of calcium solution. At 3% calcium solution, there is a decrease in the intensities of smectite (from 63% to 38%) and illite (from 12% to 8%) compared to that of kaolinite (from 25% to 20%), which decreased slightly. For a higher dosage of calcium solution (5%), the reduction in the intensity of the three minerals (smectite, illite, and kaolinite) is significantly greater than

with a 3% calcium solution (Fig. 11 and Table 5.). The addition of a calcium solution induces a reduction in both minerals, smectite and illite, and a small reduction in kaolinite, which demonstrates the reactivity of these minerals during treatment.

Regarding the treatment of sample OMB3 (Fig.12 and Table 5), it is observed that the addition of 5% calcium solution results in a reduction in the intensity of all clay minerals. The reductions in the intensity peaks of smectite (from 64% to 4%) and illite (from 13% to 3%) are significant: almost all of these minerals disappeared at a percentage of 5% calcium solution, with only the kaolinite peak persisting with a lower intensity than in the untreated sample (from 23% to 17%). In contrast, an addition of 3% calcium solution showed no reduction in the intensity of the minerals.

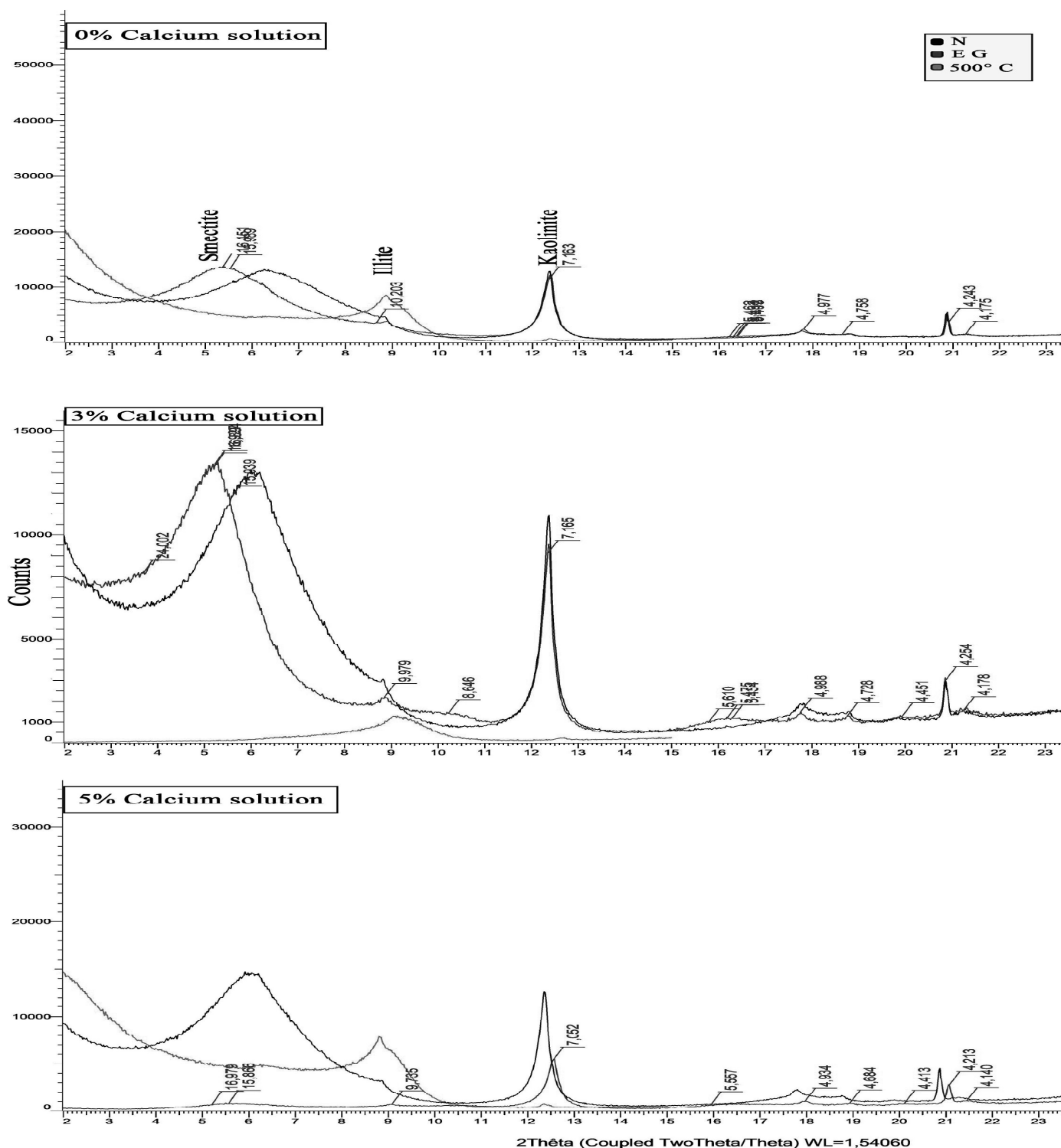


Figure 12. X-ray diffraction of an oriented slide of the sample (OMB3) untreated and treated with calcium solution.

DTA and TG thermal analyses

Thermal analyses make it possible to complete the identification by monitoring and quantifying mass losses according to the temperature ranges characteristic of the different phases.

The thermograms of sample N1 in its natural state show the existence of three endothermic peaks. The first peak is between 100 and 200°C, due to the departure of hygroscopic water from the particles surface (dehydration). The second peak between 475 and 550°C corresponds to the dehydroxylation of clay minerals (the reaction during which

the clay minerals lose their hydroxyl). The last peak, located between 700 and 800°C, corresponds to decarbonation, indicating the carbonates presence in this sample. Concerning the thermograms of the treated samples, the results show the appearance of a new peak in the temperature range of 250 to 300°C corresponding to the formation of hydrated compounds of (CSH) type; the mass loss, which is associated with the formation of these compounds, increases with calcium solution addition increasing. It should be noted that the mass loss of clay fraction minerals (smectite, chlorite, illite and kaolinite) decreases with calcium solution addition increasing Fig. 13 A.

The results of the thermogravimetric analysis are identical to the results of the XRD analysis. These observations are similar to the results reported by several studies (Maubec et al. 2017; Guidobaldi et al. 2017).

The thermogravimetric data for the natural state sample Boum2 are presented in Fig. 13B. The natural state sample shows three mass losses. The first, between 90 and 125° C, corresponds to both the desorption of water physically adsorbed on the particle's surface and the intermediate water of clay minerals. The second mass loss, located between 475 and 900 °C, is linked to the dehydroxylation of the structural OH groups of smectite, illite and kaolinite. The third mass loss, located between 775 and 825 °C, is linked to sample decarbonation. Thermogravimetric analysis of samples treated at 3% and 5% of the addition showed a rapid and complete consumption of the added stabilisers as well as a rapid formation of new hydrated phases between 250 and 300°C. It should be noted that the percentage of mass loss of the clay fraction minerals, especially of the smectite and illite, decreases with increasing calcium solution addition.

Two temperature ranges can be described for the OMB3 sample treated with 3% and 5% calcium solution addition. The first was in the temperature range of 250–350 °C, in which all processed samples had a greater mass loss than the sample in its natural state. This interval is mainly governed by the decomposition of hydrated phases such as calcium silicate hydrate (CSH), which was identified by XRD analysis. The second temperature range is that corresponding to the dehydroxylation of smectite. The absence of portlandite confirms the consumption of portlandite and the formation of new cementitious phases (CSH) during the 28-day curing period. These results are consistent with the XRD results (Fig. 13C).

In summary, we see that the results of the three treated samples are very close. The diffractogram study of total clay highlighted the reactions triggered by the calcium solution addition, showing that the reflections characteristic intensities of portlandite are consumed over time, suggesting a strong reactivity of this phase within the material. Cementitious compounds such as CSH calcium silicate hydrates are clearly detectable after 28 days of curing. The peak intensities of the clay fraction decrease with increasing added calcium solution. The reduction in the reflection of clay resulting from the reaction of this fraction with the calcium in the solution.

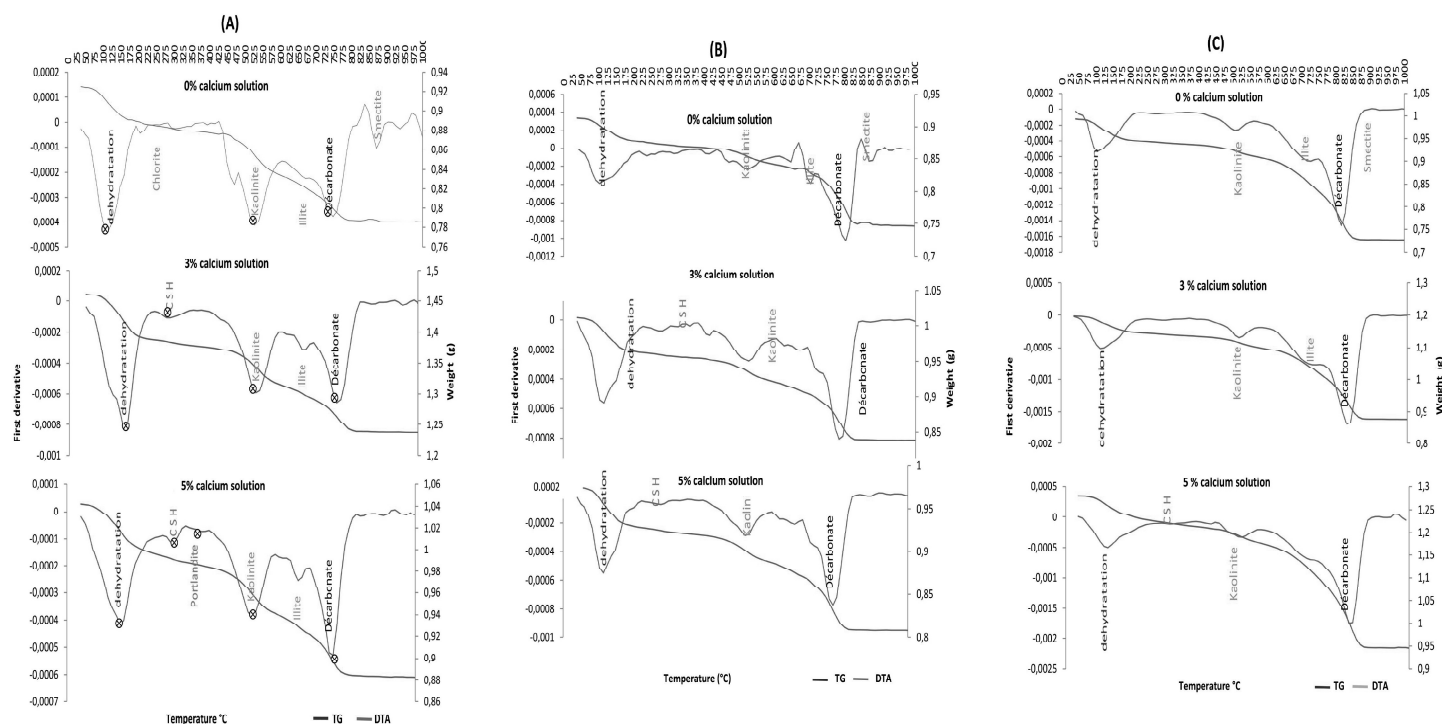


Figure 13. DTA and TG (corrige sur la figure) curves of untreated and treated samples: (A) N1, (B) Boum 2, (C) OMB3.

Effect of treatment on the microstructural of samples

Mineralogical and microstructural studies were carried out to study the mineralogy and microstructure of the samples treated with calcium solution. In particular, they aimed to determine the existence of cementing minerals and the formation of ettringite, known for its potential to cause swelling.

SEM images (Fig 15, 16 & 17) of untreated samples N1 Boum2 and OMB3, clearly show that the samples have a discontinuous structure, where the pores are more visible. Upon treatment with a calcium solution, a change in the size of the aggregates was observed. The presence of calcium ions and a high pH increase the size of the clay particle aggregates due to cation exchange reactions (reactions between the cations associated with the clay particle surfaces and the calcium cation) and flocculation. The exchange and attraction effect of the cations leads to the clay particles being brought closer together to form flocs. The immediate flocculation effect of clay particles has a more disparate particle size. The figures clearly show that the treatment resulted in a more compact structure in the treated samples (agglomeration).

The surface of the treated samples observed at large scale appears to be covered with a cement product such as calcium silicate hydrates (CSH) detected by mineralogical analysis (XDR). These products are due to the reaction of calcium with silicon and released by clay minerals since this reaction is a basic attack that promotes the progressive destruction of aluminum silicates, and its quantity increases with time. These cement products lead to greater cohesion inside the material (Al-Mukhtar *et al.*, 2012; Maubec *et al.*, 2017). Fillings of micropores by these cement products were observed and also developed inside the agglomerates (Fig. 15).

From the results of the mineralogical analyses presented in Table 1, it is observed that the three samples have a sulphate content that varies between 1 and 6%; sample N1 contains 6% sulphate while samples Boum2 and OMB3 contain proportions of 2% and 1% sulphates respectively. Based on the studies made by (Aldaood *et al.*, 2014), lime

treatment could be effective in stabilizing gypsum soils containing a small amount of gypsum ($\leq 5\%$) and in improving their swelling properties. The mineralogical results presented in (Fig. 7,8 and 9) are consistent with that reported by (Aldood *et al.*, 2014). Regarding sample N1, which contains 6% sulphate, mineralogical and microstructural studies show the appearance of cementitious minerals such as calcium silicate hydrates (CSH) were present in sample N1 treated with 9% lime (Benyahia *et al.*, 2020), with the absence of ettringite, known for its potential to cause swelling (Fig. 14). Aldood *et al.*, 2014 were interested in the swelling potential behavior of lime-treated gypsum soils, mineralogical and microstructural studies were carried out to evaluate the cementitious materials and the presence of ettringite. These authors noted two observations from X-ray diffractions. The first is that the intensities of all kaolinite reflections decrease during hardening, regardless of the gypsum content. This behavior is attributed to the fact that kaolinite is exhausted by the pozzolanic reaction and is consistent with the pozzolanic behavior of kaolinite. According to Aldood *et al.*, (2014), the main cementitious compounds were calcium silicate hydrates (CSH), calcium aluminate hydrates (CAH), and ettringite mineral. Observations of SEM images made by Aldood *et al.*, 2014 found that, on samples cured at 20 °C, no ettringite crystals were observed for any of the gypsum percentages used. Ettringite crystals start to appear at 180 days of curing and for 25% gypsum. The average length of these crystals does not exceed 5 μm . In the samples cured at 40 °C, the results are almost the same as at 20 °C but an early appearance of ettringite crystals at 28 days for 25% gypsum can be observed. In addition, the ettringite crystals appear to be larger than those observed at 28 and 180 days when the curing temperature was 20 °C. This increase in the scan size of ettringite crystals is explained by the effect of thermal activation when the temperature increases from 20 °C to 40 °C (Al-Mukhtar *et al.*, 2010).

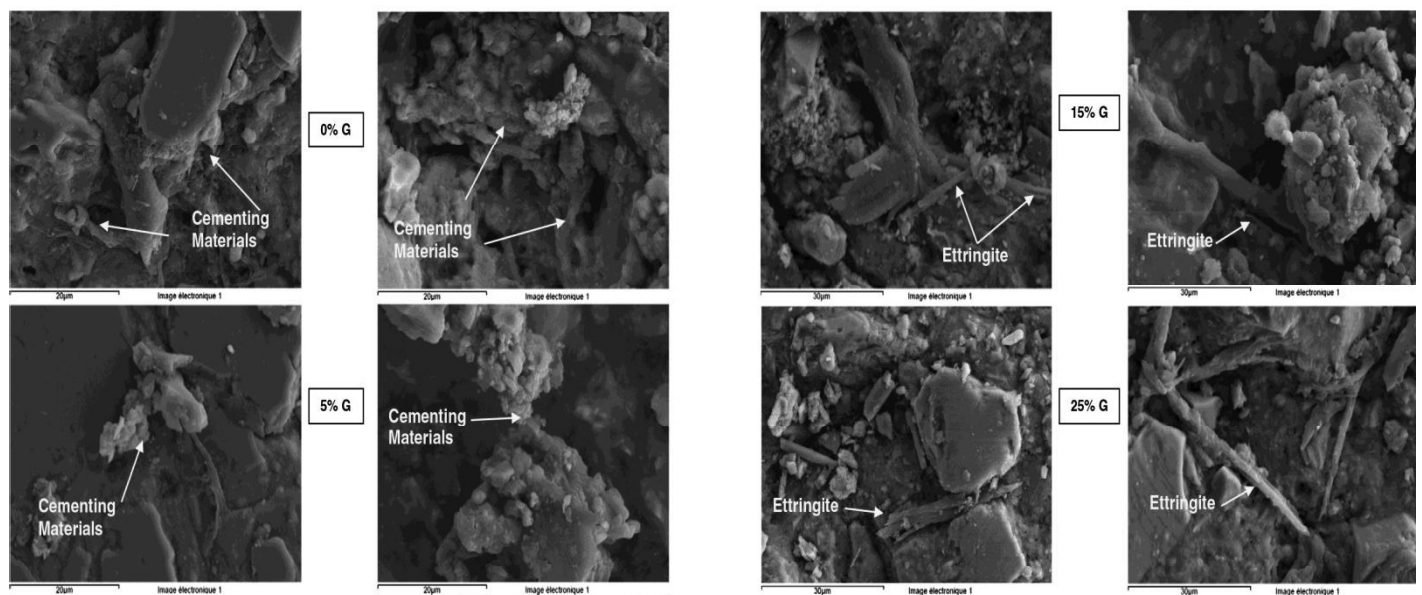


Figure 14. SEM images show cementitious materials and ettringite development for soil samples hardened at 40 °C. (Aldood *et al.*, 2014)

In our case, it is noted that ettringite crystals were not observed on the SEM images for sample N1 (Fig. 16) treated with 5% calcium solution. This behavior can be explained by the rapid infiltration of calcium in solution form inside the clay particles and formed the cementitious compounds CSH and that calcium in solution form penetrates much faster and does not give time for the formation of ettringite crystals with the presence of gypsum, which gives this

type of treatment an advantage over other treatments (for example lime treatment).

Observations by SEM images clearly show for sample N1 treated with 9% lime (Benyahia *et al.*, 2020) (Fig. 15) the absence of ettringite, a mineral that forms in alkaline environments by reacting with calcium hydroxide and clay destabilization products, the absence of ettringite can be justified by the proportion of sulphate 6% and that this quantity of sulphates is not sufficient to form the ettringite mineral.

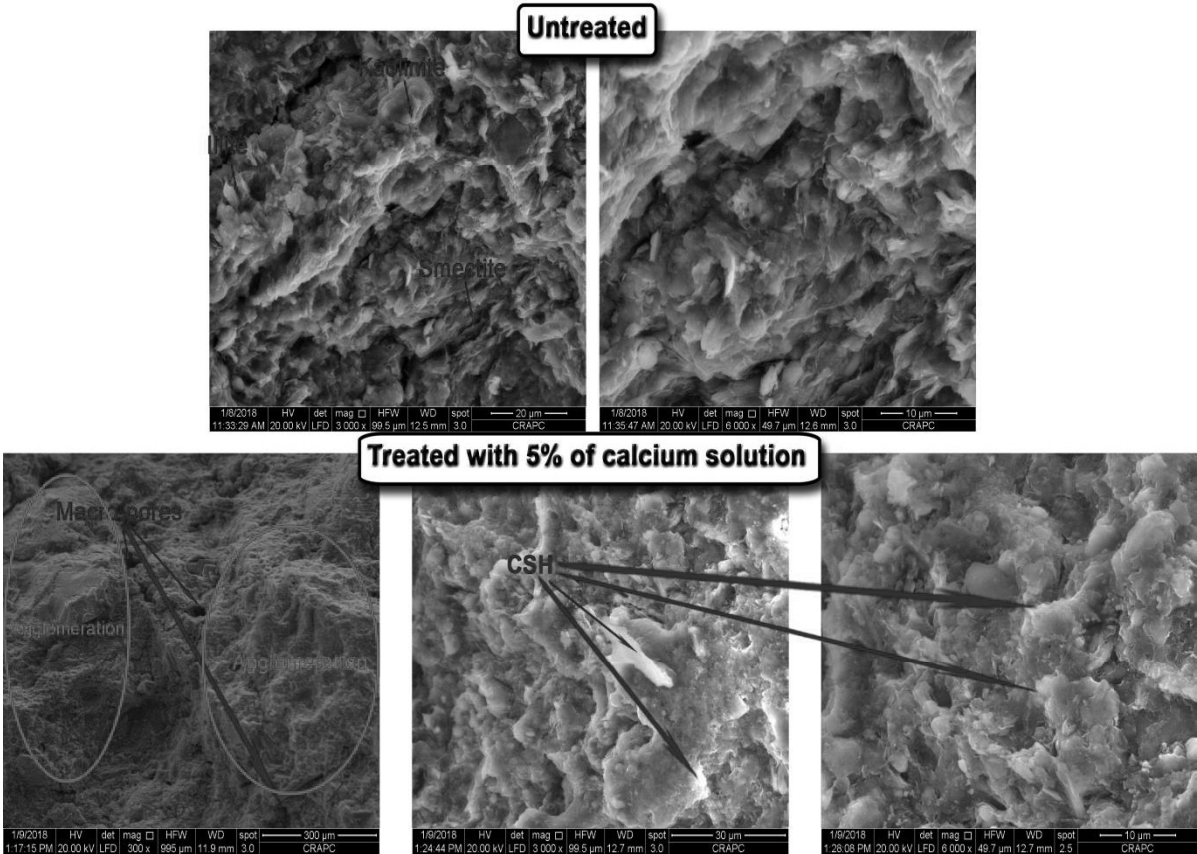


Figure 15. SEM picture of sample N1: untreated, and Treated with 5% of calcium solution

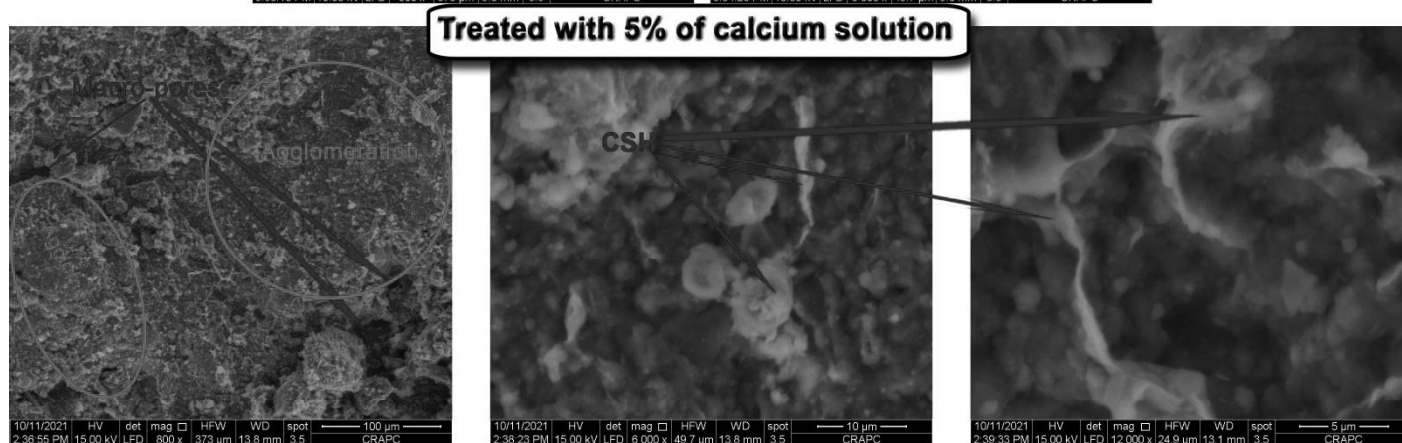
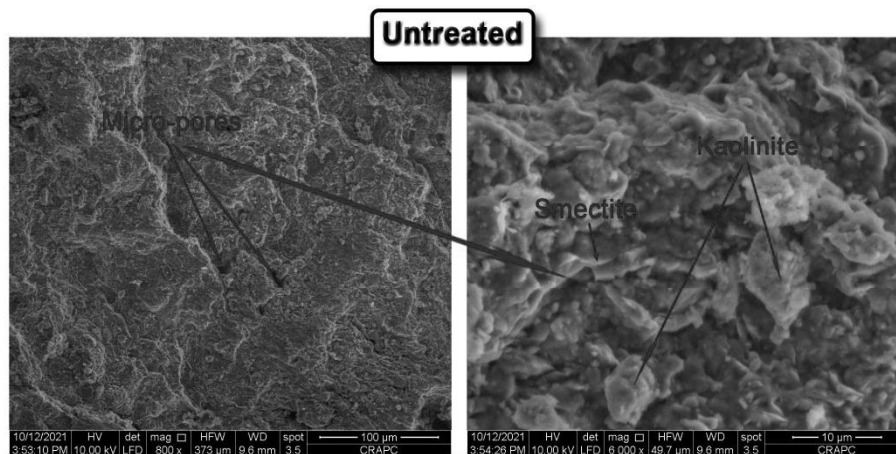


Figure 16. SEM picture of sample Boum2: untreated, and Treated with 5% of calcium solution

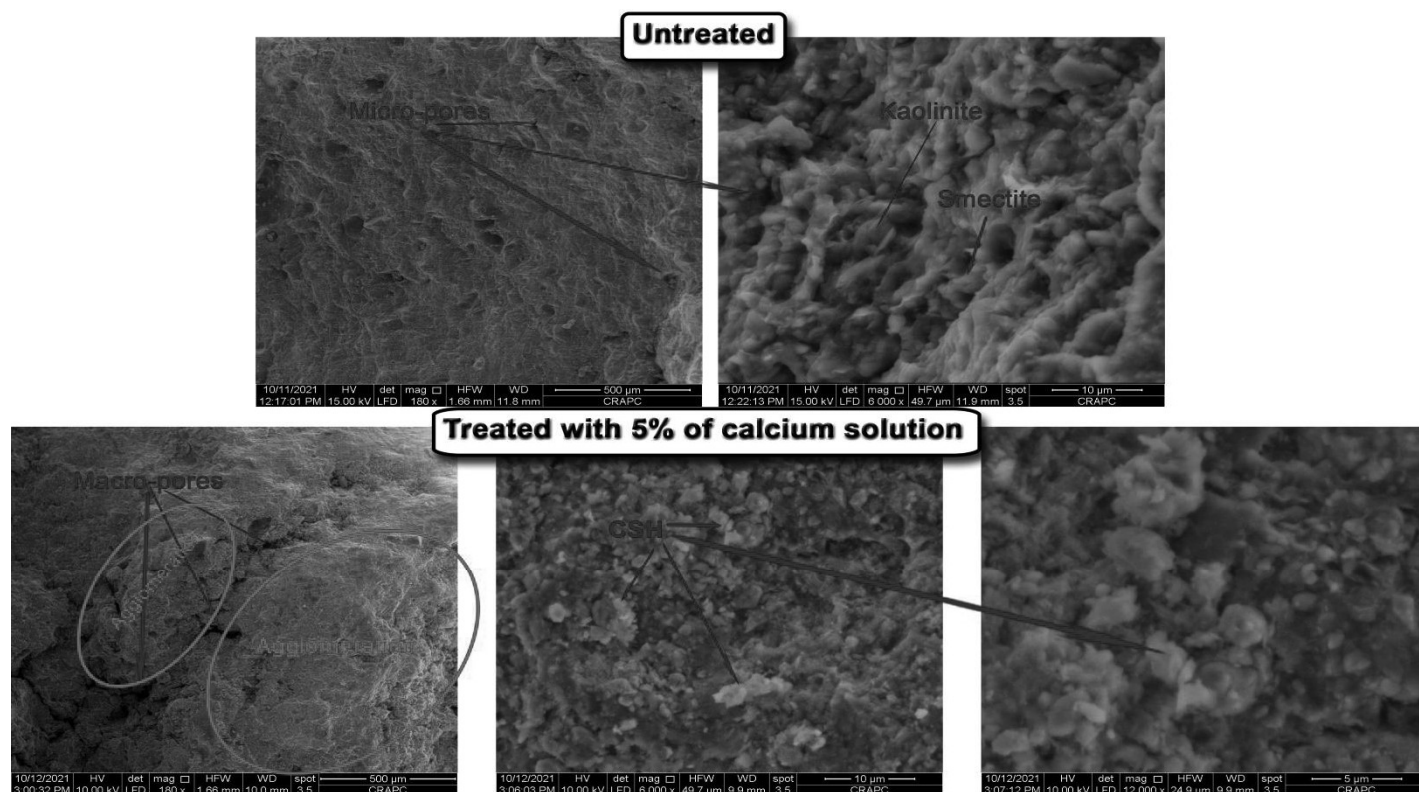


Figure 17. SEM picture of sample OMB3 : untreated, and Treated with 5% of calcium solution

Discussion

The treatment effects with a calcium solution on clays are the consequence of chemo-mineralogical reactions, namely the cation exchange between calcium ions and the clay minerals surface (smectite, illite, kaolinite, chlorite, etc.) as well as pozzolanic reactions, which develop over time due to the high availability of calcium, aluminum and silicon in the soil-adjuvant system. XRD and DTA/TG analysis demonstrated reactivity of the three samples coming from three different locations. Sample N1 is consisted of 65% clay, quartz (16%), calcite (13%) and gypsum (6%) with traces of dolomite and feldspar. Its clay fraction is made up of 26% smectites, 28% kaolinite, 15% illite, 5% interstratified (smectites+illite) and 26% chlorite. The Boum2 sample is composed of clay (65%), quartz (18%), calcite (20%), gypsum (2%) and traces of feldspar and dolomite. The clay minerals contained in this sample are mainly composed of 63% smectites, 25% kaolinite and 12% illite. Sample OMB3 contains mainly 53% clay (64% smectites, 23% kaolinite and 13% illite), 9% quartz, 34% calcite and a small amount of gypsum, dolomite and feldspar.

The diffractograms of dry and disoriented total powder of the three treated samples and for the two percentages (3% and 5%) show that there are variations in intensity and relative abundance of the clay fraction ($d = 4.46 \text{ \AA}$) during the treatment, which decreases with the addition percentage increasing, which shows that there is a reactivity of this fraction. Parallel to this reduction, we note an appearance of the reflection intensity of cementitious compounds (CSH). The reduction in the intensity of the clay fraction during treatment is due to the formation of pozzolanic type reaction products (CSH) during curing periods. These products are associated with the reaction of calcium with silicon released from clay minerals.

Regarding the intensity and relative abundance of other non-clay minerals, which did not show a clear reduction during the treatment, they indicate that these minerals were not significantly influenced by the treatment. It should also be noted that the peak quartz reduction (SiO_2) was not observed, suggesting that the quartz was not influenced by the reactions to become another source of silicon. In the OMB3 sample, we observe the increase in the calcite peak during the treatment, which is explained by the carbonation of this sample because it has a high carbonate rate (36%).

Studies carried out by (Deneele *et al.*, 2016) show that carbonates were also detected in the very short term after the treatment of silt with lime. According to (Maubec, 2010), carbonation is perceived as a reaction which disrupts the treatment because it consumes part of the calcium intended for pozzolanic reactions without improving the mechanical performance of the treated materials.

X-ray diffractograms of the clay fraction less than $2 \text{ }\mu\text{m}$ (prepared as oriented aggregates) demonstrate a different reactivity of the clay minerals, as previously indicated that there is a decrease in intensity and relative abundance of the treated clay fraction.

In the presence of minerals with cation exchange capacity in clays, calcium cations (Ca^{2+}) made available by dissolution can be exchanged with cations present in untreated minerals (Bell 1996; Al-Mukhtar *et al.*, 2010a, 2012; De Windt *et al.*, 2014; Chemedda *et al.*, 2015) determining flocculation and a modification of the particle size distribution. Also the pozzolanic activity is linked to the presence in the clays solid phase of silica and of alumina made available by the environment, induced by the presence of calcium in the water (Guidobaldi *et al.*, 2018). The evolution of pozzolanic activity is strongly dependent on clay mineralogy (Deneele *et al.*, 2016; Maubec *et al.*, 2017; Vitale *et al.*, 2016a; Vitale *et al.*, 2016b; Vitale *et al.*, 2017).

The results of XRD analysis on this fraction show that the intensities of all smectite reflections decreased during treatment. This behavior is attributed to the fact that smectite is most affected by pozzolanic activity and illite is less

influenced by the pozzolanic reaction than smectite. It should be noted that the kaolinite and chlorite peaks did not show much reduction during treatment. These results are in agreement with Eades and Grim (1966), who studied the effect of hydrated lime on several clay minerals (kaolinite, halloysite, calcium and sodium bentonite and montmorillonite). They showed that clay minerals react differently with lime and that illite and montmorillonite are more strongly destroyed than kaolinite and halloysite.

De Windt *et al.*, 2014, show that the continuous dissolution of montmorillonite during pozzolanic reactions becomes the source of silica leading to the formation of (CSH) and that the pozzolanic activity of lime-treated clays strongly depends on the mineralogy of the clay. For the treated bentonite samples, a rapid consumption of portlandite was detected for lime contents up to 20%, the high reactivity of the clay minerals smectite and illite favors the formation of CSH compounds in the very short term (Vitale *et al.*, 2017). According to Chemedda (2015), coating the kaolinite surface with a layer of calcium or $\text{Ca}(\text{OH})_2$ adsorbed on the kaolinite surface prevents dissolution reactions favored by the high pH environment, also reducing the release of silicon and aluminum into the system and delaying the kinetics of pozzolanic reaction.

Consequently, this adsorption would slow down the kinetic dissolution of kaolinite and explain a higher Ca^{2+} reactivity of bentonite. Vitale *et al.*, (2017) explain the cause of the high reactivity of bentonite by the rapid and total consumption of portlandite in the very short term, which can be attributed to the greater cation exchange capacity of the montmorillonite and its large specific surface area (Boardman *et al.*, 2001).

The differences observed between the dissolution of kaolinite, chlorite, illite and montmorillonites are generally due to a structural difference. Maubec *et al.*, (2017) show that the precipitation of secondary phases such as calcium silicate hydrate and calcium aluminate hydrate appear more clearly in the case of Ca^{2+} bentonite treatment suggesting faster dissolution of it. Vitale *et al.*, (2016a) studied the influence of pore water chemistry on the short-term behavior of lime-treated soil, according to Vitale *et al.*, (2016a) the arrangement of kaolin particles is strongly dependent on pH, with a split face-to-face arrangement at pH 4.6 which changes to a dispersion at high pH and the addition of suspended calcium ions induces the formation of larger particle aggregates, formed by stacking multiple face-to-face particles with face-to-face arrangements.

However, in the case of the swelling soils treatment with a calcium solution, it seems that the calcium ions (Ca^{2+}) have an important influence on the mineralogy of clays; because all clay minerals are almost influenced and only traces remain of smectite, illite, small amounts of chlorite and kaolinite then it is very clear in samples N1 and OMB3.

The almost disappearance of smectite type minerals with the calcium solution is probably linked to the high pH of the pore water, because the increase in pH accelerates the alteration of this mineral due to its high exchange cationic capacity and its large specific surface area. Remember that this treatment also allows an almost total reduction in sensitivity to water.

Concerning the thermograms of the treated samples, the results show the appearance of a new peak in the temperature range from 250 to 300°C corresponding to the formation of hydrated compounds of the type (CSH), the loss of mass which is associated with The formation of these compounds generally increases with increasing solution addition. It should be noted that the mass loss percentage of clay minerals decreases with increasing addition. The results of the thermogravimetric analysis are identical to the results of the XRD analysis. These observations are similar to the results reported by several studies (Maubec *et al.*, 2017; Guidobaldi *et al.*, 2017).

In summary, the main cementitious compounds found in the treated soil samples were calcium silicate hydrates (CSH),

the absence of the other compounds CAH and CASH may be due to the mineralogical nature of the clay minerals, in particular the presence of smectite, which provides silicon to form CSH compounds. These hydrated compounds formed by calcium, silicon and aluminum released by clay minerals, especially smectite considered as the main source, connect the clay grains together by forming numerous and solid bridges leading to their stiffening, and consequently, the reduction in swelling. Similar stabilization results were obtained using lime, according to studies carried out by (Benyahia *et al.*, 2020) on the same sample N1, only in this case the sample stabilizes at 5% lime addition. When lime (CaO) is added to soil, an exothermic reaction occurs within the first few hours. This involves the hydration of lime, which leads to the formation of calcium hydroxide (Ca(OH)₂).

The solubility of hydrated calcium hydroxide (Ca(OH)₂) in pure water decreases with temperature, the reaction results in the release of calcium (Ca²⁺) and hydroxyl (OH⁻) ions into solution. The latter will lead to a more or less significant increase in soil pH depending on the quantity of added lime (Maubec 2010). In our case a very satisfactory improvement of the soil with the addition of a small percentage of calcium solution, is explained by the solubility of hydrated calcium hydroxide (Ca(OH)₂) in water and the release of calcium ions (Ca²⁺), which is used directly in contact with the soil, unlike the case when lime was used.

The specificity of a treatment with a calcium-rich solution compared to a lime treatment was clearly demonstrated, that this treatment is an effective reducer of swelling pressure. The results obtained on the addition of a calcium solution to the swelling soil of these regions show that this treatment can constitute a solution to consider, because with low percentages of addition there is a significant reduction in the pressure of swelling. This use of the treatment is explained by the fact that this technique is the one which presents the maximum advantage, particularly with regard to the cost and the methods of execution compared to that of other treatment methods.

The SEM images of sample N1, Boum2 and OMB3 before any treatment show a loose structure with several open pores scattered in the field of view (Fig 16 & 17). After treatment, however, there are few pores and in fact, more cementation seems to have taken place. Consequently, the structure becomes denser, and the cementing material appears as white lines and aggregates covering the field of view. Also, the absence of ettringite crystals is noted in both samples and also for sample N1 (sample presents 6% sulphate) treated with 9% lime (Figure 15).

The SEM images of sample N1, Boum2 and OMB3 before any treatment show a loose structure with several open pores scattered in the field of view (Fig 15, 16 & 17). After treatment, however, there are few pores and in fact, cementation seems to have taken place. Consequently, the structure becomes denser, and the cementing material appears as white lines and aggregates covering the field of view. Also, the absence of ettringite crystals is noted in samples and also for sample N1 (sample presents 6% sulphate) treated with 9% lime cited by Benyahia *et al.*, 2020.

Conclusion

The results given in this article make it possible to comment on the influence of treatment with calcium solution on the swelling and on the mineralogy of clay soils, focusing mainly on the dissolution of minerals from the clay fraction, which is considered to be the main swelling factor.

It was also shown in this study that the effectiveness of this treatment on the samples swelling, which is the consequence of the chemo-mineralogical evolution in the treated materials. The changes in the geotechnical properties of soils resulting from the addition of a calcium solution are due, on the one hand to the dissolution of clay minerals which are considered to be the main factor in the swelling of these soils, and on the other hand to the formation of

cementitious compounds of the CSH type.

The mineralogical study by X-ray diffraction on the total powder of the treated samples identified new phases of hydrated calcium silicate type (CSH). The results of the XRD analysis on the clay fraction of the treated samples show that the formation of a cement phase of the hydrated calcium silicate type (CSH) is related to the presence of smectite in the clay minerals of these soils.

It was found that the treatment reduced the intensities of clay minerals, including smectite, which was greatly influenced compared with illite, which was less influenced. On the other hand, kaolinite and chlorite showed a little reduction. The characterizations carried out by thermal analysis show the formation of new CSH type phases from the dissolution of clay minerals.

Mineralogical and microstructural studies reveal changes in the microstructure of the treated samples, a decrease in the number of pores, the structure becomes denser and the cementing material covers the aggregates. We also note the absence of ettringite crystals in both samples and especially for sample N1 (the sample presents 6% sulphate) treated with 9% lime.

The results obtained from the addition of the calcium solution to the swelling soil of these regions show that this solution works very satisfactorily and can constitute a solution to consider because with low percentages (2%) of addition, there is a significant reduction in swelling pressure.

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