1st IMEKO TC-4 International Workshop on
Metrology for Geotechnics
Benevento, Italy, March 17-18, 2016

The contribution of microstructural investigation to the study of lime-treated soils

Marta Di Sante[1]

[1] *Department of Materials, Environmental Sciences and Urban Planning, Technical University of Marche, Via Brecce Bianche, 12 Ancona - Italy, m.disante@univpm.it*

***Abstract* – It is widely known that lime treatment of clayey soils enhances mechanical and hydraulic characteristics of soils. In soil-lime systems, this improvement is due to a sequence of chemical processes that results in reaction products detectable at meso- and micro-scale. Therefore the contribution of experimental techniques such as Scanning Electron Microscopy, Energy Dispersive X-ray Spectroscopy and Mercury Intrusion Porosimetry turns out to be essential to analyze the results of geotechnical tests and to understand the behaviour of soil-lime mixtures. After a brief review of literature on the microstructure of lime stabilized soil, the paper presents the results of research studies carried out on clayey soils treated with lime by means of the previous cited techniques.**

I. INTRODUCTION AND BACKGROUND

Lime treatment of clayey soil is a widespread and versatile technique. The mechanical and hydraulic improvement in soil-lime systems is due to a series of chemical processes. Given that reaction products are detectable at a meso- and micro-scale, the contribution of experimental techniques such as Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Spectroscopy (EDS) and Mercury Intrusion Porosimetry (MIP) turns out to be essential to study the lime-treated soils. Microstructural investigation can help to analyze the results of geotechnical tests and the mechanical and hydraulic behaviour of soil-lime mixtures.

Reviewing the literature on lime treated soils, comments on experimental results are often helped by microstructural investigations.

The use of SEM to investigate soil-lime mixtures became widespread in the second half of the 90s; works from Locat et al. [1] and Rajasekaran et al. [2] are early examples of the application of this technique at soil-lime mixtures. Also the paper of Bell, that developed a huge following in the field of lime treatment of soils (more than 450 citations attracted), was published in 1996 [3], SEM equipped with energy dispersive spectrometer is used to identify the differences in the reaction products between lime-treated kaolinite and montmorillonite. The study of Bell was helped by results of X-Ray diffraction (XRD) too. A later contribution from Rajasekaran and Narashima Rao [4] includes the convenient use of SEM techniques to identify the formation of new products as a result of lime induced changes in a marine clay in order to comment on its permeability characteristics. Typical pozzolanic products and fabric changes induced by cation exchange can be usefully detectable by SEM (e.g. [5], [6])

Also XRD analyses offer a big contribution to identify the formation of new crystalline phases in different soil types such as expansive clays or gypseous soils [7], [8]. XRD technique also allows to study the influence of lime amount [9], curing temperature [9],[10] and soil composition [11] on the soil-lime reaction products.

MIP offers an insight into the material structure by describing the pore size distribution of small samples. Locat et al. [1] used MIP to compare treatments with different amount of lime and to evaluate the effect of consolidation processes. Variations in pore size distribution can be observed as a function of the moulding water content and of curing time too [12], highlighting the reduction of cumulative pore size with reactions progress. Also the study of the combined presence of lime and gypsum in soils can be performed by means of MIP [8].

Aim of the present paper is to show several applications of micro-scale investigation in the field of lime treatment of clayey soils by analyzing:

- the influence of the clay mineralogical composition, determined by XRD, on consistency limits of several lime-treated soils;
- the effects of a delay in compaction (i.e. prolonged mellowing) on soil-lime dry unit weight with the help of SEM observations and EDS;
- how curing condition in terms of water content can affect both the development of reaction products and the hydraulic conductivity of soil-lime samples, commenting on geotechnical test results by using SEM, EDS and MIP .

A. Soil-lime chemical reactions

The addition of quicklime (CaO) to any type of soil-water system improves soil workability as a result of hydration of quicklime (exothermic) to form hydrated lime, $Ca(OH)_2$. This process is named “soil drying”.

$Ca(OH)_2$ immediately dissociates into the pore water of soil in Ca^{++} and OH^-, causing pH to raise. In clayey soils only, this dissociation results in a sequence of reactions widely reported in the literature [13], [14].

Ion exchange of Ca^{++} for the existing cations of clay (K^+, Na^+, H^+ etc.) reduces particles double-layer thickness, (helped by the increasing electrolyte concentration in the pore water [15]) and mineral particles re-arrange in an edge-to-face configuration with intra-aggregate porosity. As a consequence, workability further improves, water affinity and the swelling potential decrease [11] and hydraulic conductivity increases [16]. The reaction mechanisms, described so far, produce the so called “lime modified soils”.

The long-term changes in soil properties are due to pozzolanic reactions that occur between calcium ions and the silica or alumina of the lattices of clay minerals. These reactions improve strength and compressibility of soils (e.g. [17],[18]). Soil-lime systems in which pozzolanic reactions developed are named “lime stabilized soil”.

B. Structure of lime treated soil

As stated by several authors dealing with this research topic (e.g. [16]), soil-lime mixtures are characterized by different levels of structure and of corresponding porosity. This configuration is due to the reactions described in section A. In Fig.1 the three levels of structure and porosity are schematically depicted.

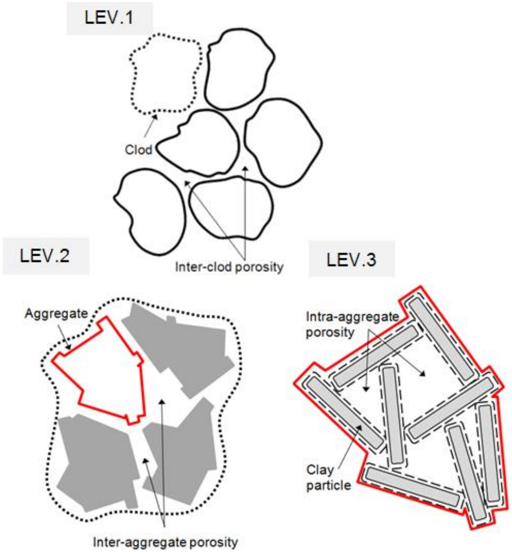


Fig. 1. Schematics of the three levels of soil structure and porosity

In Level 1 (observable with the naked eye) clods of aggregates are depicted, between them, inter-clod porosity arises. Level 2 focuses on one clod that is a group of aggregates with the corresponding inter-aggregates porosity. Level 3 is related to clay particles arranged in the edge-to-face configuration forming an aggregate, between particles there is the intra-aggregate porosity.

II. MATERIALS AND TEST METHODS

The soils used for the experimental test programs are those listed in Table 1. They are natural clayey soils of high and low plasticity.

Table 1. Characteristics of tested soils (w_L=liquid limit; w_P=plastic limit; PI=Plasticity Index).

	TESTED SOIL (Abbreviations)						
characteristic	**MON**	**SGT**	**TOR**	**FOS**	**PVB**	**PVG**	**PIA**
sand (%)	16	3	4	10	12	10	5
fine (%)	84	97	93	90	88	90	95
clay (<2μm,%)	44	39	34	32	47	38	25
w_L (%)	62	40	49	42	55	50	52
w_P (%)	30	20	27	24	24	22	30
PI (%)	32	20	22	18	31	28	22
USCS	CH	CL	CL	CL	CH	CH	CL

Also two artificial bentonites (sodium bentonite and calcium bentonite) are used for the study of the effects of lime on plasticity.

The lime used in the research is a fine calcic hydrated lime, classified as CL-80 (UNI EN 459-1), completely passing through the ASTM 200 sieve (75 μm opening). The quantity of lime added is 5% by dry mass of soil.

For the study of the effect of lime on plasticity, consistency limits (ASTM D4318-10) are also determined after lime addition. For this purpose, lime and soil were mixed at the dry state, then distilled water was added to reach the desired consistency and, after 24 hours since the binder addition, consistency limits are determined.

All the specimens for the other tests are prepared by mixing soil and lime at the wet state (as usually done in the field (e.g. [19]) and by compacting the mixture according to ASTM D698-12 (Standard Proctor Dynamic Compaction) except for the number of layer : only one layer instead of three to achieve a more homogeneous density across the sample.

For the study of the effects of compaction delay, the mixture was divided in two portions: one was immediately compacted (sample named “i”) and the other one was stored for 48 hours at room temperature in a sealed container; at the end of the mellowing period it was compacted with the same procedure of sample “i” (sample recorded as “r”). Both samples were tested at the same curing time.

In order to investigate the effect of curing conditions (in terms of water content) on the development of reaction products, only samples compacted close to the optimum moisture content (wopt) were tested. Some of them were cured in submerged (wet) conditions while other were cured at a constant water content (i.e. in sealed containers).

The permeability tests were carried out in flexible walls permeameter with tap water as permeant; tests are started varying the time spent in unsaturated conditions (0-2-7 days). In some cases, confining stress was increased during the test (from 35kPa to 380 kPa).

Microstructural investigations were carried out by means of:

- Philips diffractometer (PW1730 X-ray generator, PW 1050/70 goniometer and CuKα radiation); the mineralogical composition of the clay fraction was estimated (to the nearest 0.1) through semi-quantitative analysis of the diffraction spectra.
- Philips XL20 Scanning Electron Microscope (W filament, maximum voltage: 30 kV), after air dewatering of samples and their gilding by means of Emitech K550 sputter coater. EDS was performed by 20-30 kV voltage.
- Micromeritics, Mercury Intrusion Porosimeter Auto Pore III (60,000 psi pressure limit, 120 volts).

III. LIME MODIFICATION: EFFECTS OF MINERALOGY OF THE UNTREATED SOILS ON THE PLASTICITY OF SOIL-LIME MIXTURES

With reference to lime modified soil (i.e. short term reactions), lime addition usually causes an improvement in workability due to the reduction of the plasticity index, PI. PI is given by the difference between the liquid, w_L, and the plastic limit, w_P. With lime addition, value of w_P usually increases, while wL does not always decrease. In Table 2 consistency limits, determined before and after 5% lime addition, are listed for natural soils (Table 1) and bentonites, together with data retrieved from literature (grey background in Table 2). The liquid limit reduces in 11 cases out of 19 (in bold in Table 2), and increases in 8 cases out of 19, not contributing, in the latter way, to the reduction of plasticity index. In some cases (5 out of 19) this compromises the reduction of the plasticity index (PI in red in Table 2).

If the mineralogical composition of the clay fraction, determined by means of X-Ray Diffraction, is taken into account, it is possible to observe that both the literature data and the experimental results show that, when swelling clays are absent or present in small amount, the plasticity index can increase. In fact, the 5 cases of increase of PI are related to: pure Kaolinite, pure Quartz, Upper boulder clay (mainly Kaolinite), Tropical soil 1 (mainly Kaolinite and Quartz) and PIA soil, that exhibits only 8% of montmorillonite.

Table 2. Variation of consistency limits before and after 5% lime addition – Experimental results and literature data (grey background)

		Soil			**Soil+5%lime**		
Source		w_L (%)	w_P (%)	I_p (%)	w_L (%)	w_P (%)	I_p (%)
Experimental determinations (bentonites)	**MON**	62	30	32	**55**	33	22
	TOR	49	27	22	**44**	31	13
	FOS	42	24	18	**41**	30	11
	PVB	55	24	31	57	35	22
	PVG	50	22	28	56	37	19
	PIA	52	30	22	57	31	26
Experimental determinations (bentonites)	**Sodium B.**	320	60	260	**273**	77	196
	Calcium B.	160	56	104	**155**	63	92
Bell,1996	Upper boulder clay	30	14	16	40	22	18
	Tees laminated clay	58	26	32	**52**	33	19
	Kaolinite	72	41	31	98	42	56
	Montmorillonite	100	62	38	**83**	69	14
	Quartz	52	38	14	71	45	26
De Brito Galvao et al.,2004	Tropical Soil 1	35	21	14	38	24	14
	Tropical Soil 2	58	30	28	**53**	31	22
Guney et al.,2007	**70% Bent+30% Kao**	385	35	350	**290**	53	237
	30% Bent+70% Kao	168	28	140	**147**	42	105
	Turkmen clay	115	45	70	**103**	50	53
Thompson,1967	AASHO road test	25	14	11	27	22	5

IV. EFFECTS OF COMPACTION DELAY

The research (carried out on MON soil, see Table 1) pointed out that a delay of 48 hours between mixing with 5% $Ca(OH)_2$ and compaction causes a systematical and significant reduction of the dry unit weight (Fig.4).

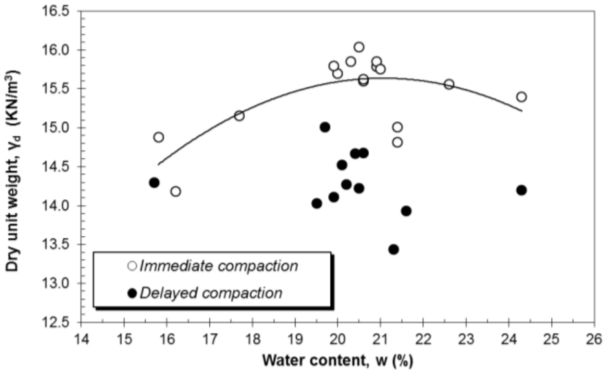


Fig. 4. Standard Proctor curves for immediate and 48-hour delayed compaction for $Ca(OH)_2$ stabilization

Although the care taken in mixing and compaction phases, delayed compaction (black points in Fig. 4), makes impossible to draw the typical trend of compaction curve for that samples. The reduction of the dry unit weight does not seem to be related to the water content.

No significant difference in the macro-structure were observed between the samples compacted immediately and those compacted with delay. SEM observations (Fig.5) allowed to verify that aggregation of particles occur in both the "i" and "r" samples as a result of cation exchange but the size of the aggregates in samples compacted with delay is higher than that of immediately compacted samples. Given that, in the "r" sample, the size of pores are bigger, too.

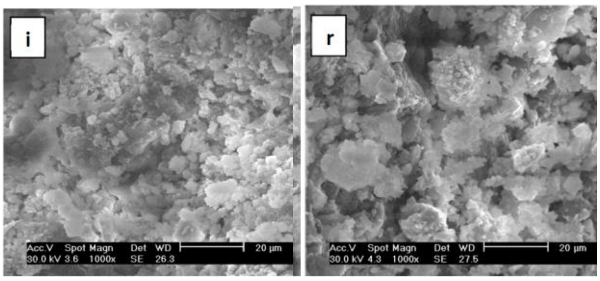


Fig. 5. SEM photos of "i" and "r" samples 2 days after the addition of lime (mod. From Di Sante et al, 2015)

This difference, detectable in the microstructure only, can be due to the pozzolanic reactions already occurred in the "r" samples, during the period of 48 hours spent at loose state. These reaction products, that bind the aggregates together, offer an higher resistance to compaction, causing a greater porosity in the sample compacted with delay. With reference to dry unit weight, quicklime treatment causes similar reduction [20].

V. EFFECTS OF CURING CONDITIONS

C. Reaction products

The microstructure of soil-lime mixtures cured in submerged conditions and that of mixtures cured at constant water content, w, were analysed using SEM supported by energy dispersive X-Ray spectroscopy (EDS). Two soil types, MON and SGT (see Table 1) are considered for the study. As shown in Fig.6, the clay particles of the untreated soil are in the typical laminated state (Fig.6a) while fully developed aggregates of particles are detectable in the samples treated with 5% $Ca(OH)_2$ (Fig.6b,c).

In the treated samples cured at w constant (Fig.6b), the nodular structure does not seem to be uniformly distributed; both zones with reaction products (R) and zones with features similar to untreated sample (non R) are detectable. This evidence, fully documented with EDS in the two different areas, for the MON soil [21][21], is verified for the less plastic clayey soil too (SGT).

It is therefore confirmed that, in partially saturated samples (MON+5%lime Sr=80%; SGT+5%lime Sr=70%), reaction products distribution is non-uniform. This can be due to the fact that water is the necessary element for the reaction between lime and soil.

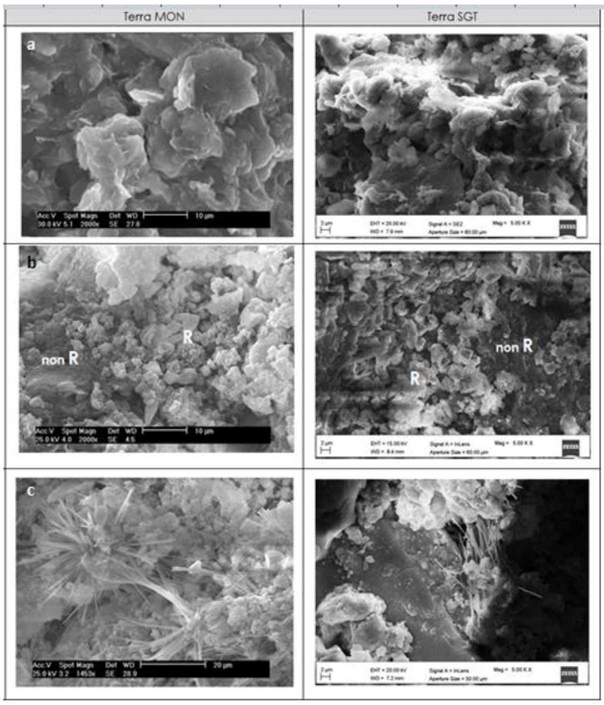


Fig. 6. SEM images of (a) untreated soils, (b) treated soils cured at constant w, (c) treated soils cured in wet conditions (submerged)

If we consider the sample at the same curing time (7days) but cured in wet conditions (Fig.6c), reaction products turn out to be more uniformly distributed across the entire sample both in amorphous and in crystal form (calcium silicate hydrate needles). Acicular structures of almost 10 μm in size develop from aggregate crumbs up to obtain bundle of CSH fibres that can connect the faces of the pores (Fig. 6c). Calcium, silicon and aluminium were detected at significant contents by EDS performed on the acicular structures.

D. Hydraulic conductivity

Also hydraulic conductivity, k, is affected by curing conditions in terms of water content (Fig.7). A general increase in k values caused by lime addition is well detectable for all the samples. In particular, with reference to the first 28 days of curing (confining stress = 35 kPa), hydraulic conductivity decreases by increasing the time spent in unsaturated conditions (0-2-7 days). In addition, the specimen cured for 7 days at constant w (black points) showed a decreasing trend in permeability and a final k value significantly lower than the other specimens (more than 2 order of magnitude).

Applying to the other two specimens an increase in confining stress during the test (from 35 to 380 kPa), appreciable reductions of k is observable. At the end of the test, the sample saturated after 2 days since the addition of lime shows a reduction of 2 orders of magnitude in hydraulic conductivity and a slight reduction in void ratio (Δe=0.06). Conversely, the k values of the sample saturated immediately showed smaller variation (within one order of magnitude); its reduction of void ratio (due to the increasing confinement) is similar to that of the other sample. Therefore, for the studied mix, an immediate saturation can assure high k value, even at high confining stress.

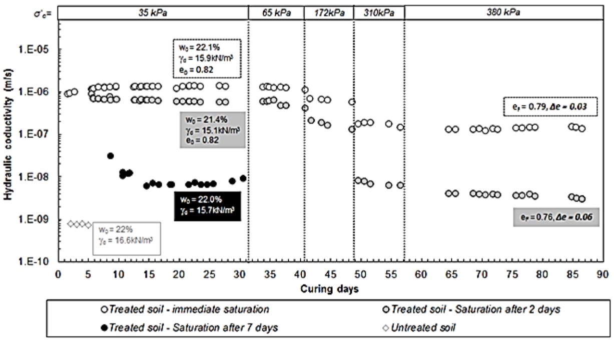


Fig. 7. Results of permeability tests carried out on specimens saturated after different curing time by varying the confining stress, σ'$_c$

Given the similar reduction in void ratio, Mercury Intrusion Porosimetry (MIP) was performed (Fig.8 and 9) in order to verify if the different behaviour could be due to a different pore size and/or to a different pore distribution.

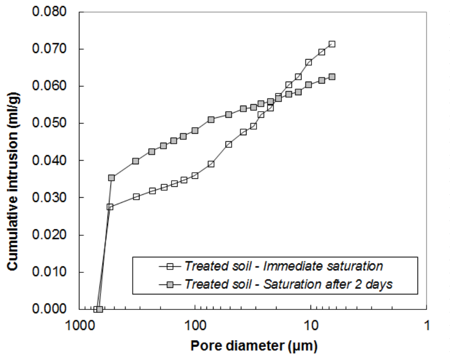


Fig. 8. MIP results -Cumulative volume intruded

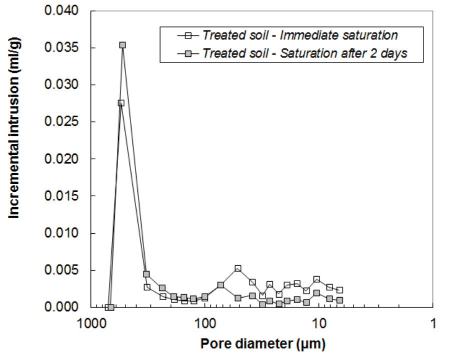
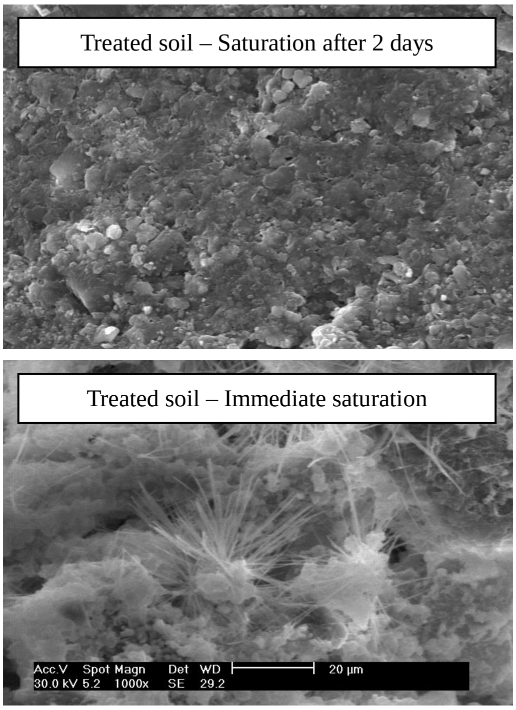


Fig. 9. MIP results - Incremental volume intruded

The MIP test pressure did not exceed 206 kPa to avoid sample crumbling, therefore only the inter-aggregate porosity was investigated. As shown in Fig.7 and in Fig.8, MIP does not highlight great differences between pore size distributions (bi-modal) of the two samples.

Also calculation of tortuosity factor from porosimetry data [22] was performed in order to evaluate if tortuosity could cause the difference in final hydraulic conductivity values. The tortuosity factors were found to be similar and equal to 2.112 for the immediately saturated sample and 2.123 for the sample saturated after 2 days. Therefore the small difference in tortuosity values of inter-aggregate porosity could not be the reason of the huge difference in the final hydraulic conductivities.

Consequently, a detailed analysis of the samples microstructure was performed by means of Scanning Electron Microscopy (SEM). SEM images (Fig.10) show that particles aggregation and pozzolanic products in gel-form are present in both the samples, but, also in this case, needle shaped crystalline pozzolanic products [23] were detected only in the immediately saturated sample.


Treated soil – Saturation after 2 days

Treated soil – Immediate saturation

Fig. 10. SEM Images of immediate saturated sample and sample saturated after 2 days taken at the end of the permeability test

These compounds, after prolonged curing, are able to keep the microstructure open (also at high confining stresses) causing the high values of hydraulic conductivity.

VI. CONCLUSIONS

The sequence of chemical processes typical of soil-lime systems give rise to reaction products detectable at mesoand micro-scale. The microstructure investigation is therefore necessary to analyse the behaviour of soil-lime mixtures and the relevance of factors that can affect this behaviour.

In the research works presented, X-Ray Diffraction allowed to identify the effect of clay mineralogy on the consistency limits of lime modified soils; Scanning Electron Microscopy helped to verify the cause of the reduced dry unit weight of soil-lime mixtures compacted with delay, by offering an insight into the microstructure. With reference to the influence of curing conditions, for the two clayey soil considered, it is possible to conclude that immediate wetting of samples is necessary to obtain a more uniform reaction between hydrated lime and soil minerals with formation of crystalline pozzolanic products, detected by SEM observations and Energy Dispersive X-Ray Spectroscopy. These compounds, after prolonged curing, are able to keep the microstructure open (also at high confining stresses) causing the high values of hydraulic conductivity.

The task of academic research is to offer useful instruments and to make research results available to practitioners and professionals. In the case of ground improvement by lime stabilization, the microstructure investigation is an essential tool for the researchers to successfully complete this task.

REFERENCES

[1] J.Locat, H. Tremblay, S. Leroueil "Meachnical and hydraulic behaviour of a soft inorganic clay treated with lime", Can. Geotech. J., vol.33, 1996, pp.654-669.

[2] G. Rajasekaran., S. Narasimha Rao, "The microstructure of lime-stabilized marine clays" Ocean Eng., vol. 24, No.9, 1997, pp.867-878.

[3] F.G.. Bell, "Lime stabilization of clay minerals and soils", Eng. Geol., vol. 42, 1996, pp.223-237.

[4] G. Rajasekaran., S. Narasimha Rao, "Permeability characteristics of lime treated marine clay" Ocean Eng., v.29, , 2002, pp.113-127.

[5] P. Beetham, T.A. Dijkstra, N. Dixon, "Lime diffusion and implications for lime stabilization practice", Comp. of Papers from the Transp. Res. Board 93rd annual Meeting, Washington DC, USA, 2014.

[6] A. Wilkinson,A. Haque, Kodikara J, "Stabilisation of clayey soils with industrial by-products: Part A", Proc. Inst, Civ. Eng Ground Improv., vol. 163, No. 3, 1996, pp. 149-163.

[7] A. Lasledj, M. Al-Mukhtar, "Effect of hydrated lime on the engineering behaviour and the microstructure of highly expansive clay", Proc. of the 12th IACMAG, 2008, pp.3590-3598.

[8] A. Aldaood, M. Bouasker, M. Al-Mukhtar, "Geotechnical properties of lime-treated gypseous soils", Appl Cl. Sci., vol.88-89, 2014, pp. 39-48.

[9] M. Al-Mukhtar, A. Lasledj, J. F. Alcover, "Behaviour and mineralogy changes in lime treated expansive soil at 20°C". Appl. Cl. Sci., vol.50, 2010, pp. 191-198

[10] M. Al-Mukhtar, A. Lasledj, J. F. Alcover,"Behaviour and mineralogy changes in lime treated expansive soil at 50°C". Appl. Cl. Sci., vol.50, 2010, pp. 199-203

[11] S.K. Dash, M. Hussain, "Lime stabilization of soils: reappraisal", J. Mater. Civ. Eng., vol.24, 2012, pp. 707-714.

[12] G. Russo, S. Dal Vecchio, G.. Mascolo, "Microstructure of a lime stabilised compacted silt", Exp. Unsat. Soil. Mech., vol.112, 2007, pp.49-56.

[13] Transportation Research Board "State of the art report 5: Lime stabilization – Reactions, properties, design and construction", Washington, USA, 1987.

[14] D.I. Boardman, S. Glendinning, C.D.F. Rogers "Development of stabilisation and solidification in lime-clay mixes." Géotechnique, vol.50, No.6, 2001, pp.533-543.

[15] J.K. Mitchell, "Fundamentals of soil behavior", Wiley, Berkley, USA, 1976.

[16] P. Beetham, T. Dijkstra, N. Dixon, P. Fleming, R. Hutchinson, J. Bateman "Lime stabilisation for earthworks: a UK perspective", Proc. Inst, Civ. Eng. – Ground Improvement, vol.168, No.GI2, 2015, pp.81-95.

[17] T.C. De Brito Galvão, A. Elsharief. G. Ferreira Simões, "Effects of lime on permeability and compressibility of two tropical residual soils." J. Env. Eng., vol.130, No8, 2004, pp.881-885.

[18] N.C. Consoli, L.S. Lopes Jr, K.S. Heineck, "Key Parameters for the Strength Control of Lime Stabilized Soils". J. of Mat. in Civ. Eng., vol. 21, No.5, 2009, pp. 210-216.

[19] E. Fratalocchi, I. Bellezza, M. Di Sante, E. Pasqualini, "Mix-design, construction and controls of lime stabilized embankments", Proc. 17th Int. Conf. Soil Mech. and Geotec. Eng., Alexandria, Egypt. Vol.3, 2009, pp.2248-2251.

[20] M. Di Sante, E. Fratalocchi, F. Mazzieri, V. Brianzoni, "Influence of delayed compaction on the compressibility and hydraulic conductivity of soil-lime mixtures", Eng. Geol., vol.185, 2015, pp. 131-138.

[21] M. Di Sante, E. Fratalocchi, F. Mazzieri, E. Pasqualini, "Time of reaction in a lime treated clayey soil and influence of curing conditions on its microstructure and behaviour ", Appl. Clay Sci., vol.99, 2014, pp.100-109.

[22] S.C. Carniglia, "Construction of the tortuosity factor from porosimetry." J. Catal., vol.102, 1986, pp. 401-418.

[23] H.F.W. Taylor, "Cement chemistry." 2nd ed. Thomas Thelford ed., 1997, London, 45.