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Chemical and physical identification and classification of an artificial soil (circular economy)

Luís Andrade Pais[1*], *Luís Ferreira* Gomes[1], *Paulo* Carvalho[1] and *Victor* Cavaleiro[1]

[1]Faculty of Engineering, University of Beira Interior, GeoBioTec, Calçada Fonte do Lameiro, 6201-001 Covilhã, Portugal

Abstract. The perception and explanation of changes in the mechanical behaviour of contaminated soils are associated with physical classification and chemical composition. Elements are evolutionary in this type of soil and in the context of a contaminated environment. Let's start by evaluating a group of artificial soils. The matrix is a granitic residual soil with a mixture (M) of hydraulic lime with used lubricating oil. The samples used have proportions of 5, 10, 15 and 20% of M in the residual granitic soil. A soil sample with 5% oil (OS5) was also made, which constitutes the optimal oil content in the soil. Afterwards, the alteration of the physical-chemical identification and the evolutionary classifications of these artificial soils were studied. The structure of the soil is altered, and this is associated with the flocculation of fine particles by the effect of lime and oil and will probably influence the mechanical behaviour of the soils. In chemical terms, it is assumed an increase in the leaching index, a decrease in the chemical mobility index and an increase in losses when it comes to artificial samples. These indices show some potential in the assessment of pollution, but eventually need adjustments, mainly in the scales.

1 Introduction

Concerns about environmental degradation have increased since the mid-1960s with greater attention to problems of air quality, water and land use planning. The last few years bring to the discussion the soil pollution and the harmful effects on the environment. However, there is difficulty in determining the contaminant and its interaction with the physical and chemical properties of residual soils and others.

The formation of granitic residual soil occurs in an open system. Weathering factors control the solubility and leaching conditions of chemical substances from the decomposition of the main minerals. The chemical decomposition of granite causes the quartz to maintain the particles somewhat altered, while the first to be altered by the action of water are feldspars and micas, producing clay minerals [1]. Hydrolysis is the main weathering process for aluminosilicates, such as feldspars. The ionized water (OH^- and H^+) dissolves the mineral forming silicon acid (H_4SiO_4). The increase in the concentration of H^+ in the water makes the

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[*] Corresponding author: ljap@ubi.pt

hydrolysis more efficient and if there is aluminium available in the decomposed mineral and under favourable physical conditions, kaolinite is formed. Minerals such as kaolinite ($H_4Al_2Si_2O_9$), illite ($KAl_3Si_3O_{10}(OH)_2$) and montmorillonite ($3Na_{0,66}Al_{2,66}Si_{3,33}(OH)_2$) are formed by hydrolysis.

The granitic residual soil when mixed with lime and lubricating oil becomes more complex when it is in contact with contaminants. Those changes are produced in the density of solid particles, in physical and chemical indices. In this new framework there is a change in the factory in a new equilibrium with probable changes in the mechanical behaviour. The granulometric and chemical evolution exists with the establishment of geochemical indexes of a quantitative and physical nature of the soil that must be verified and evaluated together with the mineralogical nature. One can use the leaching index, β, mobility index, Imob [2], red hot losses, PR [3] and the different molecular relationships [4]. This information can help to understand the mechanical behaviour of the different soils used.

2 Samples

The natural granitic residual soil was investigated as a matrix for the mixture of used lubricating oil and lime. The mixture obtained is composed of lime (C) and used lubricating oil (O) in an optimal concentration of components so that an exothermic reaction occurs in order to correct the pH and neutralize the heavy metals contained in the used oil [5]. Artificial soils are produced using 5% to 20% of the mixture in natural soil. Thus, two groups of samples were formed: i) granitic residual soil with various proportions of mixtures (It's the artificial soils): M5 to M20; ii) granitic residual soil with 5% lubricating oil: OS5. The content of 5% of oil to be added to the soil was chosen because the lubricating effect decreases significantly for higher values.

The visual appearance of the material from the artificial samples and the natural soil sample with different proportions of oil is shown in Fig. 1.


AMOSTRA CA-1 TIPO MISTURA
DESCRIÇÃO ÓLEO+CAL+H2O
PROCEDIMENTO SECA NA ESTUFA
LOCAL COVILHA DATA DE COLHEITA 29/11/03
PROCESSO/ENSAIO / MISTURA+SOLO

AMOSTRA LMP-15 TIPO MISTURA
DESCRIÇÃO SOLO ø<4,75 + MISTURA 15
PROCEDIMENTO SOLO + 15% MISTURA
LOCAL COVILHÃ DATA DE COLHEITA 25/11/03
PROCESSO/ENSAIO / Proetor LMP

a) b) c)

Fig. 1. Artificial samples: a) composite mixture with optimal concentration of OS5 components; b) artificial sample M15; c) sample of natural soil with different proportions of oil.

3 Identification and classification

3.1 Particle Size Analysis

The granulometric analysis process in these soils is problematic. They are soils with a set of particles of different minerals and different degrees of alteration, which makes it difficult to individualize them. It is associated with the contingency that clayey products are flocculated and very difficult to separate. However, different separation procedures were used in the sedimentation phase (use and absence of sodium hexametaphosphate as deflocculant) which little or nothing changed the percentages of clays and silts (increase of 1 to 3% of clay in tests using sodium hexametaphosphate).

Contaminated soil shows a decrease in the coefficient of uniformity, an increase in the percentage of gravel and sand and a marked decrease in the diameters belonging to the definition of clay and silt. The results of the remaining soils reveal some similarity that indicates that they are well graded soils, as verified by the high values of the uniformity coefficient (47.5 <CU <200). The high values may not be associated with the disposition of the particles in natural conditions, intact and in situ, but with the treatment and testing process.

The identification and classification tests on the soil were carried out in accordance with the procedures described in the British Standards Institution [6]. The granulometric curves of the artificial samples with 5, 10, 15 and 20% of artificial mixture compared to natural soils and contaminated with 5% of used lubricating oil, are presented in Fig. 2.

Comparative granulometric curves of natural soil and artificial soils reveal that the addition of the mixture significantly alters the original size of the particles. Fine soil particles agglutinate due to the effect of lime and oil and form nuclei of lime hydrate in flakes of greater dimensions, as shown in the images obtained, Fig. 3, by backscattered electrons.

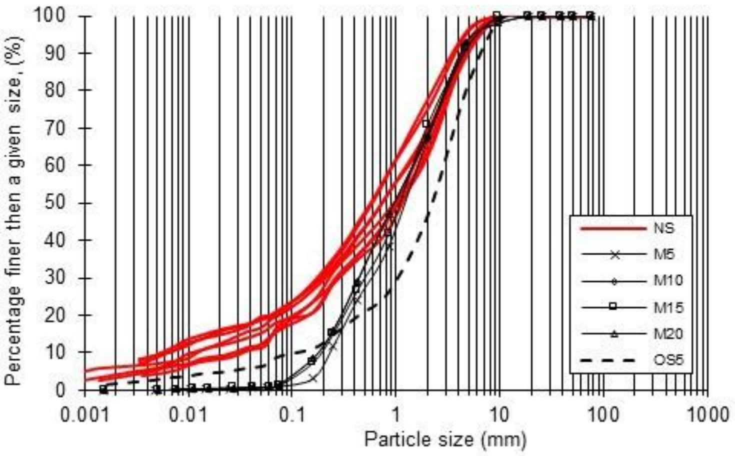
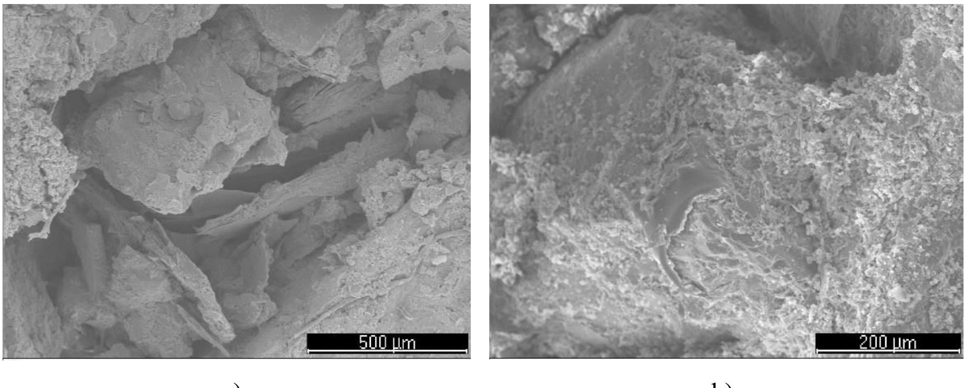


Fig. 2. Comparative granulometric curves of the GRS natural soil with the artificial soils M5 to M20 and OS5.



a) b)

Fig. 3. Images obtained by electron backscattering: a) natural soil; b) artificial soil M10.

In these artificial soils, the percentages of clay and silt practically disappear due to the agglutination of fines and the percentage of sand and gravel is similar between them. The coefficient of uniformity and the coefficient of curvature decreases. The granulometric distribution seems to be independent of the mixture proportion used in the soil (5 to 20% of lime+oil). In the OS5 soil, the distribution tends towards characteristic values of granulometric distribution closer to the natural soil. It could mean that the combined effect of lime and oil becomes important in the agglutination of fines.

3.2 Consistency Limits and Classification

Liquidity limits were obtained for the representative samples by the cone method (English) due to difficulties in estimating the closure of the soil furrow in the Cassagrande shell. The liquidity limit is obtained by the intersection of the horizontal corresponding to the 20 mm penetration with the regression line of the various points obtained. They are preferably located in a range of 15 to 25 mm of penetration. Fig. 4 presents the test results for different types of artificial soils compared to natural soil.

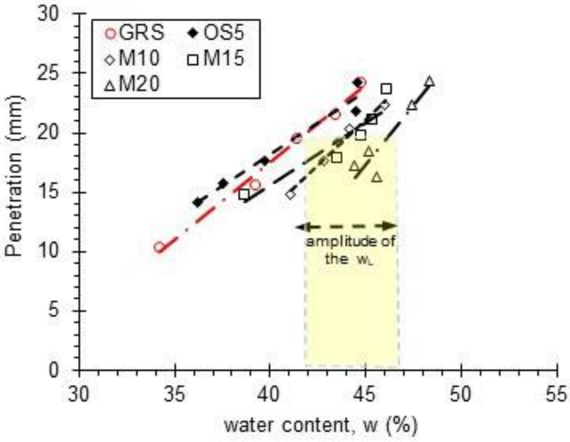


Fig. 4. Determination of w_L based on the cone test (BS, 1990): b) natural and artificial soil.

Typical values for the liquid limit (w_L) of granitic residual soil can approach 40%. They reflect the influence of the high percentage of mica and feldspar, which cause water retention in the internal cleavage planes, and not their plasticity, which is low with clays that are not very expansive in their composition. The results are consistent with those obtained for w_L by the Cassagrande method, with the results in all samples significantly higher by 4% to 5% and with the same degree of variation in all samples tested.

The values obtained for the w_L in the different artificial soils deviate from the obtained results for the standard soil in an amplitude of 4 to 5%, which is related to the alteration of water retention in the internal planes of the feldspars and micas or even by the modification of the ion exchange capacity of the clays present. In artificial soils, w_L increases with increasing mixing ratio. The effect of lime in reducing the affinity for water is contrary. Probably because it was previously mixed with lubricating oil.

The granitic residual soil of Covilhã is always characterized by well-graded granulometric curves and with low plasticity indexes. The thin ones are classified as ML due to their low plasticity. The artificial soils are classified as ML but with a slight tendency towards the virtual classification of OL.

Natural granitic residual soil by soil classification [7] belongs to the SW-SM group with gravel or silty sand with gravel and normal to low clayey activity which indicates the presence of kaolinite, which is a clay with little expansion. The classification of soils undergoes a change when it comes to artificial soil due to changes in the granulometric composition and associated plasticity index, as it can be seen in Table 1.

Table 1. Characteristics of plasticity, clayey activity and classification of artificial soils.

Sample	liquidity limit w_L(*) (%)	Plasticity Index PI (%)	Clay activity At	Classification
M5	43.0	4.6	-	SW-SP with G
M10	44.0	11.6	-	SW-SM with G
M15	44.0	10.8	-	SP to SM
M20	46.5	12.7	-	SW-SM with G
OS5	30.5	11.5	-	SW with G

(*) w_L calculated by the English cone method. The relation for Casagrande comes: $w_{Lcp}=w_{Lcc}+5$

The plasticity index is low in natural soil because it is linked to the low ion exchange capacity of the fines present (1.53 and 1.90 mE/mg). Plasticity index grows by decreasing the plasticity limit (w_P) in artificial soils. The oil replaces the water holding capacity of the fines and increases the lubrication of the particles. The calculation of clay activity also gives us an indirect assessment of the type of clay present (kaolinite).

4 Chemical characterization - natural and artificial soils

The contents of Li_2O, Na_2O, K_2O, Be_2O, MgO, CaO, TiO_2, Cr_2O_3, MnO, Fe_2O_3, CoO, NiO, CuO, ZnO, CdO, Al_2O_3, SiO_2, PbO, P_2O, H_2O and red losses in natural and artificial soils were evaluated.

The result of the chemical analyses in percentages of oxides from the natural soil (GRS), the mixture (M* and M** - lime+lubricating oil), the granitic residual soil mixed with 5% of used lubricating oil (OS5) and the soils with 5% and 15% of M (M5 and M15), are shown in Table 2.

Table 2. Chemical composition of natural granitic residual soil samples and artificial samples constituted from them.

Oxides	Sample					
	GRS %	M* %	M** %	OS5 %	M5 %	M15 %
Li_2O	70 ppm	34 ppm	36 ppm	55 ppm	85 ppm	83 ppm
Na_2O	0.97	0.03	0.03	1.07	1.24	1.27
K_2O	5.12	0.06	0.07	4.91	4.81	4.32
Be_2O	-	-	-	-	-	-
MgO	0.42	0.25	0.33	0.37	0.65	0.61
CaO	0.08	53.78	53.90	0.10	2.51	6.64
TiO_2	0.44	0.05	0.07	0.42	0.44	0.39
Cr_2O_3	87 ppm	7 ppm	-	114 ppm	116 ppm	62 ppm
MnO	137 ppm	33 ppm	39	131 ppm	161 ppm	161 ppm
Fe_2O_3	2.68	0.12	0.13	2.83	2.47	2.57
CoO	20 ppm	20 ppm	21 ppm	-	-	-
NiO	38 ppm	26 ppm	22 ppm	32 ppm	47	37 ppm
CuO	-	-	-	-	14 ppm	-

Oxides	Sample					
	GRS %	**M*** %	**M**** %	**OS5** %	**M5** %	**M15** %
ZnO	106 ppm	268 ppm	258 ppm	52 ppm	86 ppm	98 ppm
CdO	12 ppm	13 ppm	16 ppm	-	8 ppm	8 ppm
Al_2O_3	18.24	0.17	0.34 ppm	16.74	17.34	16.01
SiO_2	69.73	0.22	0.11	67.11	64.98	60.21
PbO	-	-	-	-	-	-
P_2O_5	0.12	0.07	0.06	0.13	0.11	0.10
Red-hot loss	3.01	44.94	44.21	5.60	5.11	8.11
H_2O	1.02	0.23	0.93	1.31	1.35	1.31

(-) searched but not detected; the samples in which the result is expressed in ppm, do not represent the oxide but the element.
(* and **) due to the difficulty in working this sample in the grinding process, two samples were made, one coarser (*) and the other less coarse (**).

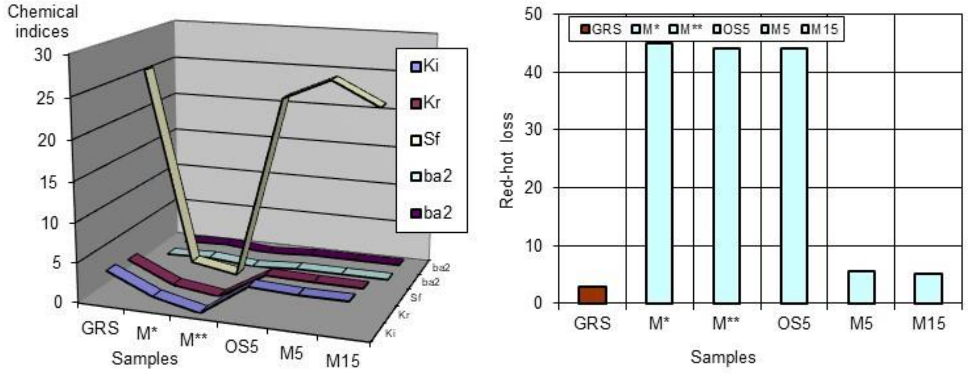
The effects of lime on natural soil characteristics are attributed to four reactions: cation exchange (general order of substitution Na+<k+<Ca++<Mg++); flocculation and agglomeration; lime carbonation (reaction of lime with CO_2 to form a relatively weak Ca and Mg carbonate cement) and pozzolanic reaction (reaction of silica and/or soil alumina with lime, being the major source of increased strength). These reactions can explain the decrease of SiO_2 and the decrease of Al_2O_3 in samples M5 and M15. Artificial soils have a decrease in the element ZnO and minor increases in Cr_2O_3 and MnO, although at the ppm level. The proposed geochemical indices for evaluating soil evolution when subjected to a mixture of lubricating oil and lime were evaluated and whose results are shown in Table 3.

Table 3. Values obtained by the geochemical indices of samples of natural granitic residual soil and artificial samples constituted from these.

Chemical alteration indices		**Sample**				
	GRS	**M***	**M****	**OS5**	**M5**	**M15**
$Ki=SiO_2/Al_2O_3$	3.823	1.294	-	4.008	3.747	3.761
$Kr=SiO_2/(Al_2O_3+Fe_2O_3)$	3.333	0.759	-	3.429	3.280	3.241
$Sf=SiO_2/Fe_2O_3$	26.018	1.834	0.846	23.714	26.307	23.428
$ba2=(K_2O+Na_2O)/Al_2O_3$	0.334	0.529	-	0.357	0.348	0.349
$b=Al_2O_3/Fe_2O_3$	6.805	1.417	-	5.915	7.020	6.229
β (leaching index)	0.681	1.079	-	0.728	0.711	0.712
Imob (mobility index)	0.370	-4.49	-4.510	0.379	0.127	-0.25
Red-hot loss	3.01	44.9	44.21	5.60	5.11	8.11

(-) researched, but it is impossible to calculate.
(* and **) due to the difficulty in working this sample in the grinding process, two samples were made, one coarser (*) and the other less coarse (**).

Fig. 5 shows the variation of these indices and the red loss for the granitic residual soil and the comparative evolution for the mixture M* and M** and for the artificial soils OS5, M5 and M15.



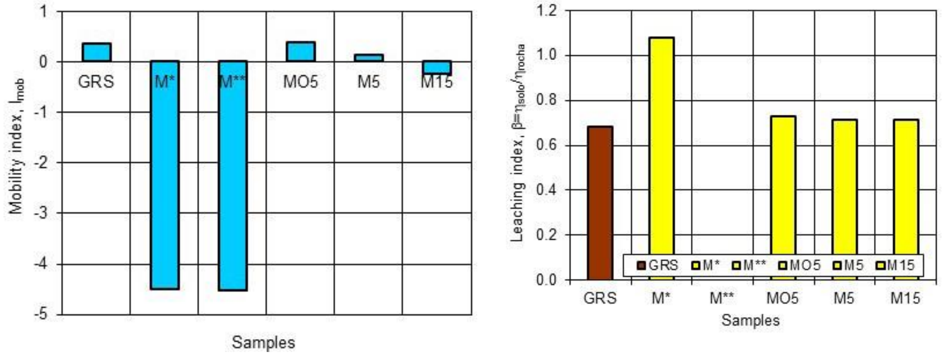
a) b)

Fig. 5. Relationship between the geochemical indices for the natural and artificial residual soil samples: a) molecular relationship; b) red-hot loss.

The molecular ratios Kr=$SiO_2/(Al_2O_3+Fe_2O_3)$ and Ki=SiO_2/Al_2O_3 for samples OS5, M5 and M15 have a slight tendency to be constant and equal to the natural sample (GRS) which may mean the maintenance of Al_2O_3 contents and Fe_2O_3. The indices b=Al_2O_3/Fe_2O_3, ba2=$(K_2O+Na_2O)/Al_2O_3$ and Sf=SiO_2/Fe_2O_3 have strong relationships in the different samples. Samples M* and M** have negligible values because they are predominantly composed of lime and lubricating oil.

The red-hot loss parameter is slightly higher in the artificial samples. The results reflect independence of the mixture proportion used because it does not suffer appreciable alteration when it comes to the M5 sample or the M15 sample. For M** (oil+lime) and OS5 (soil+5% oil) the increment is substantial and similar in both cases. It allows us to conclude that the increase in red hot loss will be associated with the lubricating oil used with lime and not with any alteration at the mineral level.

The mobility index (Imob) decreases for artificial soils and with increasing proportion of the mixture in the soil. The leaching index (β) reveals the decrease in leaching of chemical constituents to the artificial samples and remaining generally constant with the increase in the proportion of M in the soil. The use of these indexes can be masked by the high levels of CaO and other levels used in these indexes, combined with the presence of chemical products in the oil used in the mixture. Figures 6 a) and b) show the relationship between the geochemical indices for the residual soil samples and the artificial samples.



a) b)

Fig. 6. Relationship between geochemical indexes for natural and artificial residual soil samples: a) mobility index; b) leaching index.

The solid granite rock, the natural soil (GRS), and the mixture (M15) [15% of (lime+lubricating oil)] were analyzed in terms of limit values (mg/kg) of heavy metals by application of Directive 86 /278/EEC [8]. The result of chemical analyzes in this type of artificial soil indicates the presence of heavy metals or organic chemicals with lower and acceptable concentrations in the agreed classifications and standards. Table 2.6 7 presents the admissible values from official documents and Table 4, results observed in chemical analyzes of the rock and soils used.

Table 4. Limit values (mg/kg) of the concentration of heavy metals obtained for GRS, M15 and granite soils.

Element	**Sample**		
	Granite	**GRS**	**M15**
aluminium (Al^1) % (mass)	3.31	4.72	2.57
arsenic (As^3) mg/kg	0.44	14.73	8.15
cadmium (Cd^2) mg/kg	1.80	0.13	0.13
chromium (Cr^1) mg/kg	8	17	9
copper (Cu^1) mg/kg	5.6	3.4	9.3
iron (Fe^1) % (mass)	0.56	1.46	0.73
manganese (Mn^1) mg/kg	82	204	58
nickel (Ni^1) mg/kg	6	32	29
lead (Pb^2) mg/kg	3.6	18.6	0.5
zinc (Zn^1) mg/kg	70	98	192

1 - Atomic absorption; 2 - Graffiti chamber; 3 – Hydrides.

All results including those of the artificial soil (M15) are within the European directive. These point towards the potential of lime to stabilize the heavy metals present in the lubricating oil.

5 Conclusion

Artificial soils have lower uniformity coefficients (0.3<CU<7.9) and CU=30 for OS5 which tends to value closer to natural soil. The comparative granulometric curves of the natural soil and the artificial soils reveal that the addition of the mixture significantly changes the original dimension of the particles. Fine soil particles coalesce as a result of lime and oil.

The w_L values deviate 5% from the common soil value, increasing with increasing proportion of the mixture used in the soil. It is related to changes in water retention capacity. The IP is low. Soils are classified from SW-SP with G to SW with G for OS5.

The chemical evolution of soils was based on geochemical indices. The contents of Na_2O, K_2O, Fe_2O_3, Al_2O_3, MgO and SiO_2 undergo changes. The elements Li_2O, Cr_2O_3, MnO, NiO and ZnO that are present at the ppm level suffer alterations in the contents. They will be associated with advection mechanisms in the particles when in the presence of contaminants.

In the evaluation of the geochemical indices, the artificial soils (M5 and M15) have a slight tendency to assume similar values between themselves and the GRS sample. It may be associated with the maintenance of Al_2O_3 and Fe_2O_3 levels. The red-hot loss grows, and it is associated with the lubricating oil and not with any mineral alteration. The alteration indices, when applied, prove to be inadequate for the contaminated soils, but show that the soils have changed. The leaching index for the evaluated natural soils is in agreement with the proposed interval for an altered soil [0 (altered)< β<1 (sane)]. β parameter varies from 0.610 to 0.625, but when contaminated we have the parameter, $0.717<\beta<0.806$, which is not consistent with the classical meaning. It shows an addition of Al_2O_3 leaching.

These relations may be of potential use in the assessment of contamination, but certainly in need of adjustments.

The results presented in terms of limit values for heavy metals in artificial soil - M15 - are within the values established by the European directive 86/278/EEC. Results are associated with lime's potential to stabilize heavy metals present in lubricating oil.

Acknowledgments

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References

1. I.K. Lee, M.R. Coop, The intrinsic behaviour of a decomposed granite soil. Geotechnique **45(1)**, 117-130 (1995). https://doi.org/10.1680/geot.1995.45.1.117
2. T.Y. Irfan, Mineralogy, fabric properties and classification of weathered granite in Cornwall, Englend. Quarterly Journal of Engineering Geology **11**, 233-244 (1996). https://doi.org/10.1144/GSL.QJEGH.1996.029.P1.02
3. T. Sueoka, I.K. Lee, M. Muramatsu, S. Imamura, Gomechanical properties and engineering classification for decomposed granite soils in Kaduna district, Nigeria, in Proc. 1st International Conference on Geomechanics in Tropical Lateritic and Saprolitic Soils - 1985, Brasilia, Vol. 1, pp.175-186.
4. P. Rocha Filho, F.S. Antunes, M.F.G. Falcão, Qualitative influence of the weathering degree upon the mechanical properties of an young gneiss residual soil, in Proc. 1st Int. Conf. on Geomechanics in tropical Lateritic and Saprolitic Soils - 1985, Brasilia, Brazil, Vol.1, pp.281-294.
5. J.N. Meegoda, C. Bin, S.D. Gunasekera, P. Pederson, Compaction characteristics of contaminated soils-reuse as a road base material. Recycled materials in geotechnical applications, 195-209 (1996).
6. BSI - British Standards Institution: Methods of test for soils for civil engineering purposes, In: BS1377 (1990), London.
7. ASTM, “Classification of soils for engineering purposes”, 1989 Standards Annual Book D2487-85, Vol. 04.08, ASTM, Philadelphia, pp. 288-297.
8. Directive 86/278/EEC of June 12, 1986.