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Linear shrinkage and the methylene blue spot test in the analysis of plasticity of granulometrically stabilized laterites

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Abstract

For a soil to be granulometrically stable, it must present low plasticity and a granulometric composition that conforms with the limits regulated by standards. This article presents a study of the clay fraction activity and plasticity of three laterites from the Triângulo Mineiro region, aiming to use them in a granulometrically stabilized base. The experimental phase contained the determination of liquidity limit, plastic limit, sand equivalent and silica sesquioxide ratio, in addition to a Methylene Blue spot test, linear shrinkage, MCT classification and X-ray diffraction. The results show that the sand equivalent alone is not able to evaluate the content of impurities of the clay fraction; it needs to be performed along with the Methylene Blue spot test. Linear shrinkage proved to be an important indicator of plasticity, capable of measuring small values of plasticity. Finally, the MCT classification precisely indicated the lateritic behavior of the samples.

Keywords: laterites, granular bases, methylene blue spot test, linear shrinkage.

1. Introduction

According to Vargas (1978), naturally stabilized soils present a granulometric curve similar to the curve corresponding to well-graded soils, associated with an active fine fraction, with low expansion and low shrinkage by water. Both the granulometric component and the fine fraction activity and plasticity must be controlled, so that the soil is properly stabilized.

According to Brazilian standard DNIT 098 (DNIT, 2007), coarse graded lateritic soils used in base construction must present the fraction that passes through the 0.42 mm sieve, with LL ≤ 40%, PI ≤ 15% and sand equivalent above 30%.

As stated by Luiza (2008), one of the inconveniences of the sand equivalent test is that is that it only allows us to evaluate the quantity of fine material in the aggregate and not its content of impurities or "*clayness*."

Nikolaides *et al.*, (2007) studied samples of lime and non-lime aggregates and came to the conclusion that the Sand Equivalent test (SE) is not a reliable way to determine whether or not an aggregate is adequate for use in a pavement base. The results show that for the non-lime aggregate they studied, the methylene blue spot test and the sand equivalent test should both be performed, and in case the sand equivalent criterion is not met, acceptance or rejection must be based on the methylene blue criterion.

According to Vargas (1978), the activity of a soil's clay fraction must be calculated according to the Skempton Theory (1953), based on the soil activity index, defined by the ratio between the plasticity index and the percentage of clay (grains with diameter smaller than 2 μ).

As specified by Fabbri (1994), although the activity coefficient proposed by Skempton is determined for the clay fraction, it also has the deficiencies that come with using Atterberg limits. Moreover, these limits are determined by the fraction that passes through a 0.42 mm sieve, which normally exhibits abundant passive material, resulting in test results not showing the activity of fine material in the soil.

Fabbri (1994) proposes that the coefficient of activity of a soil's granulometric fraction be determined by the methylene blue spot test, taking into account the volume of methylene blue adsorbed by the soil and the percentage the soil contains of this fraction.

Paige-Green (1989) concludes that the linear shrinkage of aggregates used in coating layers was the most reliable indicator of plasticity for all the tested materials. While developing compaction prediction models, Semmelink (1991), as cited in Paige-Green and Ventura (1999), concluded that the linear shrinkage was one of the most significant characteristic parameters of the materials.

The general objective of this research is to analyze the plasticity and activity of the clay fraction of three laterites from the Triângulo Mineiro with the objective of using them as pavement base construction material with coarse graded lateritic soil.

From the aforementioned objective, the following specific objectives were adopted: analyze the results of the indicators of plasticity and clay level designated by standard DNIT 098 (DNIT, 2007) (LL, PI, Sand Equivalent), submit the laterites to both linear shrinkage and a methylene blue spot test, and analyze the results of these tests, such as indicators of plasticity and clay fraction activity, analyze the plasticity and activity of the mixtures of laterites with sand, and analyze the importance of using new indicators not present in standard DNIT 098 (DNIT, 2007).

2. Sand equivalent and methylene blue spot test

2.1 Sand equivalent

The Sand Equivalent is a volumetric relationship that corresponds to the ratio between the height of sand and the height of clay suspension in a certain quantity of soil or small aggregate. The test is done with a standard test tube, according to the procedures of testing method DNER-ME 054 (DNER, 1997).

Following standard DNIT 098 (DNIT, 2007), the granulometrically stabilized base using coarse graded lateritic soil and the coarse graded lateritic materials designated for base construction must present a Sand Equivalent (SE) above 30%.

According to Serra (1987), using the sand equivalent to evaluate the existence of unacceptable quantities of silt and clay in soil-aggregate mixtures in the tropics might lead to abandoning materials with good behavior in the base. He presented the results of fourteen samples of LFGS (lateritic fine-grained soils) used on roads in the countryside of São Paulo that presented SE between 2% and 28%, with a mean value of 11,1%, which were successfully used in pavement bases.

As the NCHRP demonstrates, there are controversial results and discoveries for the sand equivalent test, which, in some cases, identifies crusher fines as harmful particles similar to clay.

2.2 Methylene blue spot test

In Brazil, the studies of Casanova (1986), Fabbri & Sória (1991), Pejon (1992) and Fabbri (1994) showed that the methylene blue spot test is an efficient, fast, and cheap alternative for the characterization of soils, especially, for road applications on tropical soils, where the traditional systems are inadequate (Bonini, 2005).

The methylene blue test consists of the tritation of a soil + water suspension with a standardized methylene blue solution in a highly agitated medium. After adding a known initial quantity of dye in the soil + water solution, a drop of the soil + water + dye solution is taken and put it on a standard paper filter. If the image formed by the diffusion of the drop on paper presents a blue aura around the core, it means that there is an excess of dye and that the equivalence point has been reached. If not, dye is added to the solution until the equivalence point is reached. From the added quantity of dye, it is possible to calculate the Specific Surface (SS) and, consequently, the cation exchange capacity (CEC) of the analyzed soil (Fabbri, 1994).

Fabbri (1994) determined the Value of Blue (Va) and the Coefficient of Activity (CA) using Equations 1 and 2. In Equation 1, Va corresponds to the total volume in milliliters of Methylene Blue consumed by 1 g of soil, V is the total volume of Methylene Blue added to the suspension during the test in milliliters, P_{200} is the percentage of soil passing through the 0.075 mm sieve and w is the moisture content of the soil right before starting the test.

$$Va = V \times \frac{P_{200}}{100} \times \left(1 + \frac{w}{100}\right) \tag{1}$$

$$CA = 100 \times \frac{Va}{P_F} \tag{2}$$

In Equation 2, the Coefficient of Activity (CA) is calculated based on the percentage of the soil fraction, in weight, from which we aim to evaluate the activity (PF) and the Value of Blue (Va).

Fabbri (1994) defined three levels of activity for clay mineral groups, according to their coefficients of activity (CA), determined by the methylene blue spot test: a) high activity (CA > 80), which comprises the montmorillonite and vermiculite group; b) activity (11 < CA < 80), which comprises the kaolinite and/or illite group, or combinations of these with more or less active groups, as long as in proportions compatible with the coefficient of activity of the characteristic group, and c) low activity (CA < 11), which comprises the passive materials and laterized clay minerals, or combinations of these with more active groups, as long as in proportions compatible with the coefficient of activity of the characteristic group. With this data, Fabbri (1994) built an abacus with the soil percentage of clay in relation to the value of blue, which enables the evaluation of the soil Level of Activity.

Figure 1 shows the methylene blue spot test in detail.

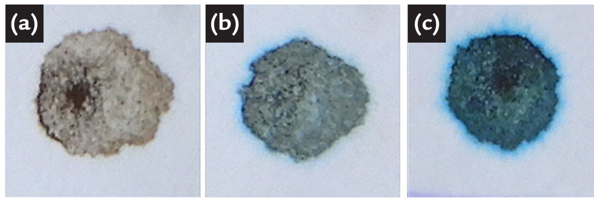

Figure 1 - Detail of methylene blue spot test, a) Start of the test on paper filter; b) Sequence, formation of the blue aura, and c) End of test after the appearance of the blue aura corresponding to the equivalence point.

2.3 Linear shrinkage

Linear Shrinkage is defined as the reduction in the length of a bar of soil, expressed as a percentage of its original length, when the soil is oven-dried in a suitable mold, from a moisture content approximately equal to the liquid limit (LL). The test is performed according to British Standard BS 1377 (BS, 1990).

According to Look (2005), as cited in Austroads (2018), volumetrically active clays are sensitive to moisture changes, which is the most significant factor when determining pavement service life and maintenance requirements. However, for Cock *et al.* (2015), as cited in Austroads (2018), linear shrinkage is an indicator capable of demonstrating these volume changes resulting from the variation in the soil content of moisture.

When deciding for the test in accordance with standard BS 1377 (BS, 1990), the fraction of soil that passes through a 0.42 mm sieve must be mixed with distilled water until the mixture soil/water reaches a consistence equal to or slightly more humid than the liquidity limit. According to standard BS 1377 (BS, 1990), this value is not critical, and a small percentage variation is acceptable. For soils that we cannot determine the liquidity limit, we must add as little moisture content as possible to enable the groove on the Casagrande device to close after 25 drops. The sample thus prepared must be placed in a metal mold of semi-cylindrical shape with a length of 140.0 mm and a diameter of 25.0 mm, lubricated with Vaseline. The mold should be placed in a sanded place until it shrinks away from the mold walls. Then, drying must be completed in an oven, first at a temperature not exceeding 65 °C until shrinkage has largely ceased, and then at a temperature of 105 °C to 110 °C to complete drying. After drying, the percentage of linear shrinkage will be calculated by Equation 3, in which L_D is the dried sample's final length (mm) and L_0 is the initial length of the moist sample.

$$CL = \left(1 - \frac{L_D}{L_0}\right) \times 100 \quad (3)$$

According to Australian standard MRWA (2022), natural lateritic gravel requires a maximum linear shrinkage of 4.0% for unaltered laterites, and a maximum of 2.0% when the material has been altered by crushing, screening or mixing. The liquidity limit should be at a maximum of 30%.

3. Materials and methods

In this study, we collected laterites from two cities in the Triângulo Mineiro. The first sample in Araguari/MG, designated as LAR3, coordinates 18° 38' 43.8"S and 48° 10' 00.0"W, the second in Coromandel/MG, designated LCR6, coordinates 18° 29' 38.2"S and 47° 14' 26.5"W, and the third in Romaria/MG, designated LRM8, coordinates 18° 54' 55.5"S and 47° 36' 08.7"W.

The samples' geotechnical characterization was done following the tests required by standard DNIT 098 (DNIT, 2007) - granulometrically stabilized base using coarse graded lateritic soil: granulometric analysis NBR 7181(ABNT, 2016c), liquidity and plastic limits NBR 6459 (ABNT, 2016a) and NBR 7180 (ABNT, 2016b), and sand equivalent DNER-ME 054 (DNER, 1997). The silica sesquioxide ratio (Kr) was obtained in accordance with standard DNER-ME 030 (DNER, 1994a).

Then, the samples were submitted to the methylene blue spot test (NBR 14949 (ABNT, 2017) standard) to evaluate the activity level of the clay fraction, to the linear shrinkage test BS 1377 (BS, 1990) and to the Mini-MCV compaction test DNER-ME 258 (DNER, 1994c), and weight loss by immersion DNER-ME 256 (DNER, 1994b) for MCT classification. The X-ray diffraction was performed at the CPMTC (Centro de Pesquisa Professor Manoel Teixeira de Castro) of the Universidade Federal de Minas Gerais (UFMG), to characterize the samples' minerals. We used a Shimadzu/LabX6000 and system θ - 2θ, with radiation CuK (λ = 1.5418Å), scanning of 5° to 80° (2θ), at a speed of 2°/min and pace of 0.02° (2θ), Reading time 0.6s. Reading voltage of 40.0 kv and Reading current of 30.0 mA. A fraction was separated that passes through a 0.075 mm sieve to prepare the samples.

Three samples of sand with laterite LRM8 were prepared to evaluate the changes in plasticity and the activity of the samples. The mixtures were submitted to granulometric analysis NBR 7181(ABNT, 2016c), liquidity and plastic limits NBR 6459 (ABNT, 2016a) and NBR 7180 (ABNT, 2016b), sand equivalent DNER-ME 054 (DNER, 1997), linear shrinkage BS 1377(BS, 1990) and the methylene blue spot test standard NBR 14949 (ABNT, 2017).

4. Results and analysis

4.1 Granulometric analysis

Table 1 and Figure 2 show the results of the samples' granulometric analysis.

Table 1 – Granulometric analysis.

Material	LAR3	LCR6	LRM8	Grading range A *	
				Max	Min
Passing # 1 (%)	97.6	94.2	84.1	100	75
Passing # 3/8 (%)	79.8	64.9	61.0	85	40
Passing # n° 4 (%)	68.4	53.8	48.1	75	20
Passing # n° 10 (%)	54.4	37.8	39.7	60	15
Passing # n° 40 (%)	50.2	33.7	33.1	45	10
Passing # n° 200 (%)	26.3	25.3	21.1	30	5
Gravel (%)	31.6	46.2	51.9		
Coarse sand (%)	14.0	16.0	8.4		
Medium sand (%)	4.2	4.1	6.6		
Fine sand (%)	23.9	8.4	12.0		
Silt (%)	10.1	8.0	4.3		
Clay (%)	16.2	17.3	16.8		

* DNIT 098/2007 standard.

LCR6 and LRM8 were classified in Grading range A of standard DNIT 098 (DNIT, 2007), but for laterite LCR6, the percentage of material passing through the 0.075 sieve mm is higher than 2/3 of the percentage of material passing through the 0.42 mm sieve, which is not allowed by the aforementioned standard. The LAR3 and LRM8 samples need a correction in granulometry to be used in base construction.

The percentage of gravel varied from 31.6% in laterite LAR3 to 51.9% in laterite LRM8. Laterite LAR3 presented the lowest clay percentage, equal to 16.2%, while for laterites LRM8 and LCR6, percentages were 16.8% and 17.3%, respectively.

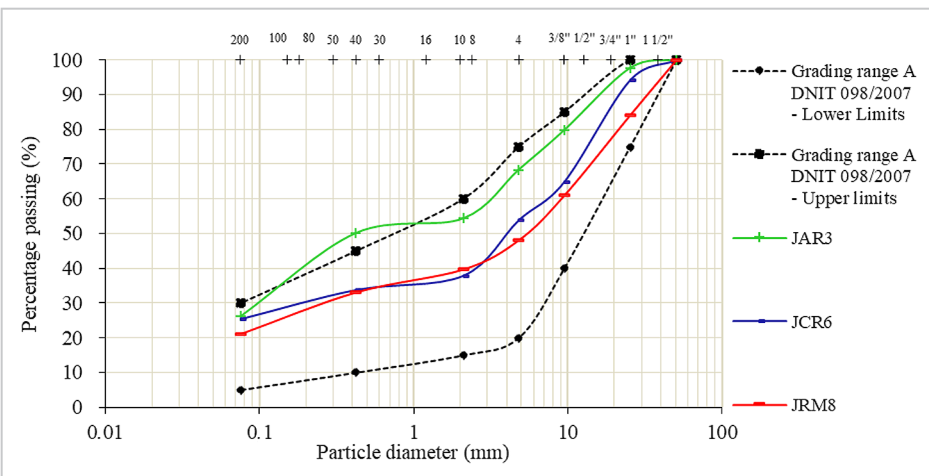

Figure 2 – Laterites granulometric curves JAR3, JCR6 and JRM8.

The curve of laterites LAR3 and LRM8 presented a plateau, comprehended between the 2.0 mm and the 0.42 mm sieves, which indicates a material deficiency in the interval. This plateau is present in the granulometric curves of many laterites that occur in Brazil.

4.2 LL e LP, Sand Equivalent, Linear Shrinkage, Methylene Blue Spot Test and X-ray diffraction

Table 2 shows the samples results for liquidity limit and plasticity index, sand equivalent, linear shrinkage, and methylene blue spot test. The values for Methylene Blue (Va) indicated on Table 2 are normalized for 1 g of clay fraction.

Table 2 – Atterberg Limits, Sand Equivalent, Methylene Blue Spot Test and XRD.

Material	LAR3	LCR6	LRM8
LL (%)	29	36	35
PI (%)	11	15	14
Sand. Equivalent (%)	16	24	16
Linear Shrinkage (%)	3.8	5.0	4.7
Va *	0.9	1.1	1.5
Activity level	Low Activity	Low Activity	Low Activity

*Methylene blue values for 1 g of clay fraction.

The three samples presented values for liquidity limit and plasticity index within the acceptable limits specified by standard DNIT 098 (DNIT, 2007). All samples presented values for the sand equivalent below 30% and, according to this criterion, should be rejected.

Figure 3 shows a graph comparing the percentage of clay with Va values. When considering the results for activity levels obtained by the methylene blue spot test, all three samples were classified on "Low Activity," indicating a small influence of clay minerals present in the laterites clay fraction.

These results show that the sand equivalent criterion is not enough to precisely indicate the level of purity of the laterites clay fraction. For these materials, when the sand equivalent criterion is not met, samples must be submitted to the methylene blue spot test, with its results overruling the ones from the sand equivalent tests.

Thus, opting for the methylene blue criterion, laterites LAR3, LCR6 and LRM8 can be used, despite their presenting sand equivalents below 30%, since their clay fraction activity level was classified as "Low Activity."

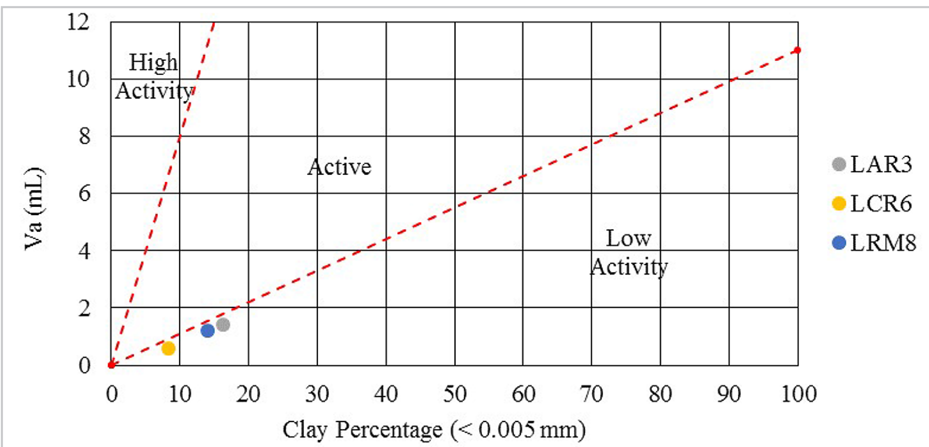

Figure 3 - Clay Percentage X Laterites Va.

Samples LCR6 and LRM8 presented linear shrinkage above 4.0%, consequently, above the recommended value for base implementation according to Australian standard MRWA (2022). Sample LAR3 presented linear shrinkage equal to 3.9%, within the limit accepted by the Australian standard.

Figure 4 shows the results for the analysis by X-ray diffraction of all three laterites. All samples indicated an extensive presence of aluminum and iron oxides (Geothite, Gibbsite, Hematite) that constitute the typical structure of laterized soils and present low activity.

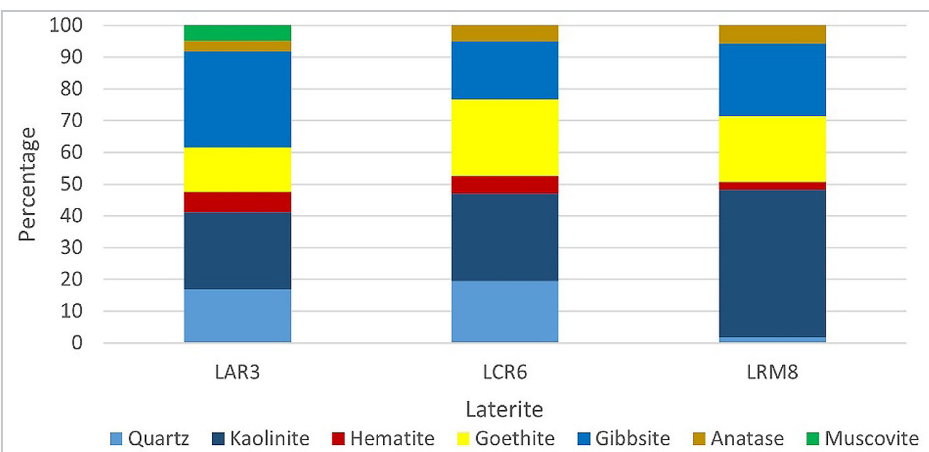

Figure 4 - Result from laterites X-ray diffraction.

In the three samples, the iron and aluminum oxides appear combined with kaolinite, which belong to the group of moderately active clay minerals. Laterite LAR3 had a small percentage of muscovite, equal to 4.9%, which normally presents moderate activity, similar to the kaolinite and illite group, but a combination of iron and aluminum oxides resulted in a material with a low activity clay fraction.

The presence of "high activity" clay minerals from the montmorillonite group was not identified and the results of the X-ray diffraction confirmed the ones obtained from the methylene blue spot test.

4.3 MCT classification, TRB classification and silica sesquioxide ratio

Table 3 shows the samples results from the MCT classification, the TRB classification and the Silica Sesquioxide Ratio. According to the TRB classification, laterite LAR3 was identified as soil A-2-6 (clayey sand) and laterites LCR6 and LRM8 as A-2-4 (silty sand).

Table 3 - Laterites MCT classification, TRB classification.

Material	LAR3	LCR6	LRM8
e’	0.72	0.85	0.94
c’	1.53	1.71	1.68
MCT Classification	LA’G’	LG’	LG’
TRB Classification	A-2-6	A-2-4	A-2-4
Silica Sesquioxide Ratio (Kr)	1.08	1.12	0.72

According to the MCT classification (Nogami & Villibor, 1995), all three laterites were identified as lateritic soils LA’G’ (sandy clay with lateritic behavior). LA'G' soils are situated near the limit between LA' soils (sandy lateritic) and LG' (clayey lateritic), and, since they have relatively high percentages of sand, they generally present properties similar to LA' soils, such as high bearing capacity under proper compaction, low shrinkage by moisture loss and low expansion (Nogami & Villibor, 1995).

The highest value of c' in sample LRM8, equal to 1.46, indicates a higher relative percentage of sand in this sample in relation to the others (14% clay and 7.4% fine sand). Laterite LAR3 presented the highest clay percentage of all three samples, equal to 16.2%, but it was associated with a percentage of fine sand equal to 23.9%, which resulted in a value of c' equal to 1.31, lower than the laterite LRM8 value of c'. Laterite LCR6 presented the lowest value of c', equal to 1.16, which indicates that this sample presents the highest relative percentage of sand (16.6% fine sand and 8.3% clay).

These results show that the MCT classification represents the real behavior of the studied laterites with more precision and more details than the TRB classification.

The three samples presented Kr values lower than two, confirming the lateritic behavior indicated by the MCT classification. There were no conflicts between the MCT classification and the X-ray diffraction. On the contrary, the minerals indicated in the X-ray diffraction confirm the MCT classification results. Finally, the "Low Activity" levels determined by the methylene blue spot test are in accordance with the LA'G' classification (sandy clay with lateritic behavior) pointed by the MCT classification. The MCT classification was necessary to indicate the lateritic behavior of all three samples.

4.4 Mixture of sand and laterite

Laterites LCR6 and LRM8 presented linear shrinkage values above 4.0% and laterite LRM8 also presented a percentage of material passing through the 0.075 mm sieve superior to 2/3 of the percentage of material passing through the 0.42 mm sieve. Due to this incoherence, three mixtures of laterite LRM8 and sand was prepared aiming to correct the granulometry and to evaluate the effect of the addition of sand in the clay fraction plasticity and activity levels. Two samples of sand were used: fine quarry sand, designated A1, and medium washed sand, designated A4. The proportions were 70% LRM8 and 30% A1, 80% LRM8 and 20% A4, and 70% LRM8 and 30% A4.

Table 4 shows the three mixtures and the test results for granulometry, liquidity limit, plasticity index, sand equivalent and the methylene blue spot test. The methylene blue values are normalized for 1 g of clay fraction.

The results in Table 4 show that mixing sand reduced the liquidity limit and the plasticity index of laterite LRM8. Fine quarry sand A1 reduced them even more, but in this case, the granulometry of mixture LRM8+A1 (70x30) did not meet the standard DNIT 098 (DNIT, 2007) Grading range A. However, it is possible to subsequently test the reduction of the fine sand component to make it fit one of the granulometric grading ranges established by the standard.

Samples LRM8+A4 (80x20) and LRM8+A4 (70x30) present granulometric distributions that conform with grading range A of standard DNIT 098 (DNIT, 2007) and the corrected distribution of fine fraction; that is, they presented a percentage of material passing through the 0.075 mm sieve lower than 2/3 of the percentage of material passing through the 0.42 mm sieve, meeting the standard's requirements. The linear shrinkage values were also reduced, and all mixtures presented values below 4.0%, meeting the standard's requirements.

Table 4 - Granulometric analysis, LL, PI, Linear Shrinkage and Sand equivalent of the granulometric mixtures.

Mixture	LRM8+A1 (70x30)	LRM8+A4 (80x20)	LRM8+A4 (70x30)
Passing # 1" (%)	94.1	94.2	89.3
Passing # 3/8" (%)	73.4	7.9	73.2
Passing # n° 4 (%)	66.6	62.9	63.6
Passing # n° 10 (%)	58.7	52.6	44.5
Passing # n° 40 (%)	52.4	3.7	24.8
Passing # n° 200 (%)	20.0	19.4	13.3
LL(%)	NL	28	27
PI(%)	NP	12	8
Sand Equivalent (%)	19	17	25
Linear Shrinkage (%)	1.0	2.9	2.4
Va *	1.0	0.9	0.6
Activity level	Low Activity	Low Activity	Low Activity

*Methylene blue values for 1 g of clay fraction.

Figure 5 presents a graph comparing the clay percentage and the Va values of the studied mixtures. All three samples clay fraction activity levels were classified as "Low Activity," indicating the samples low activity of clay minerals. The results show that adding sand reduced the laterites Va values.

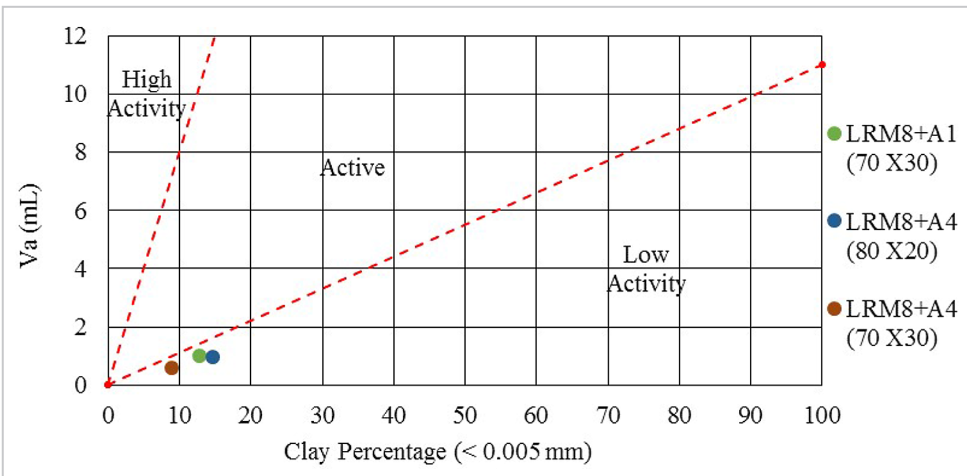

Figure 5 - Comparison of clay percentage and Va values of the mixtures.

Considering the plasticity requirements of standard MRWA (2022), the three sand mixtures presented the necessary characteristics, with liquidity limit below 30% and linear shrinkage below 4.0%. When considering the 2.0% maximum limit for linear shrinkage, only mixture LRM8+A1 (70x30) met the required value.

Considering the requirements of standard DNIT 098 (DNIT, 2007), all three sand mixtures should be rejected because they presented sand equivalents below 30%, but when applying the methylene blue criterion, all mixtures met the necessary requirements, since they were classified as "Low Activity." The liquidity limit and the plasticity index values of all three samples were also within the acceptable limits established by standard DNIT 098 (DNIT, 2007).

5. Conclusions

The three studied laterite samples presented a sand equivalent below 30%. Therefore, they did not present the sand equivalent characteristics required by standard DNIT 098-2007, and according to this requirement, all three should be rejected.

When the methylene blue criterion was associated with the sand equivalent, all samples were classified as "Low Activity," indicating that they can be used.

The three sand and laterite mixtures also presented a sand equivalent below 30%, but, when associating the methylene blue criterion with the sand equivalent, they presented the necessary characteristics to be used as material in base construction because their clay fraction activity was classified as "Low Activity" by the methylene blue spot test.

These results show that for laterites, when the sand equivalent does not meet the standard's requirements, the samples must be submitted to the methylene blue spot test. Then, we should opt for the methylene blue criterion; that is, if the methylene blue spot test indicates a "Low Activity" clay fraction, the sample can be used. On the other hand, if the methylene blue spot test indicates an "Activity" or "High Activity" clay fraction, it must be rejected.

The results show that the linear shrinkage is an important plasticity indicator that can measure low plasticity values, even below 2%, as observed in mixture LRM8+A1 (70x30). In contrast, the plasticity index cannot precisely measure low plasticity values and classifies every material in this condition as "non-plastic", NP, with no distinction among them. The linear shrinkage property will be useful in cases where it is necessary to analyze the laterites used in the base, which must present low plasticity values.

With this, it is possible to infer the importance of the linear shrinkage and the methylene blue spot test to analyze the plasticity and activity level of bases built with granulometrically stabilized laterites, since these provide information that neither the sand equivalent nor the plasticity index can.

The MCT classification was necessary to indicate the fine fraction lateritic behavior of coarse graded lateritic soils.

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