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Mechanical behavior of bitumen emulsion treated-lateritic gravels

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ABSTRACT: Laterites are tropical soils, resulting from parent rock weathering and mostly composed of SiO_2, Al_2O_3 and Fe_2O_3, met in quartz, kaolinite, goethite and gibbsite. For potential use in road base and sub-base layers, treatment is often required. The most common method for such stabilization remains the addition of hydraulic binders (cement or lime), prone to cracking and presenting a high carbon footprint. This paper aims to investigate the mechanical properties of laterite treated with bitumen emulsion. To consider the influence of the petrological nature of the lateritic gravels, three different materials from Côte d'Ivoire and Cameroon were analyzed in terms of Atterberg limits, compaction (modified Proctor) and California Bearing Ratio (CBR) index, completed by the chemical composition and mineralogical structure assessment. To evaluate the bitumen emulsion effect on laterite stabilization, two surfactants at 1% content by weight of emulsion and two bitumen emulsion contents (3% and 5% by weight of laterite) were selected. The treated laterites were assessed using indirect tensile strength (ITS) and stiffness modulus tests after 7 and 28 days of curing at 35 °C and 80% relative humidity. The results highlighted the influence of laterite nature on its mechanical properties. Furthermore, the critical role of surfactant chemistry was observed: one of the tested emulsions demonstrated a high affinity for laterite whatever its origin, contrary to the second one. The increase in bitumen emulsion content enhanced lateritic mechanical characteristics, emphasizing the action of bitumen as a cohesion agent. Thus, the addition of bitumen emulsion on raw laterite may improve its properties and constitutes a viable solution for the durability of roads in a tropical context.

KEYWORDS: Laterite, bitumen emulsion, surfactant, mechanical characterization, stiffness modulus.

1 INTRODUCTION

Laterite soils are formed by parent rock alteration due to tropical climatic conditions. During the laterization process, weathering causes the leaching of silica in the parent rock, followed by an increase in iron and aluminum oxides. The sum of these species can reach 92±5% (Kumar et al., 2022). Laterite is chemically classified into two distinct categories: ferralitic and ferruginous. The most widely used criterion for this classification considers the silicon oxide content. With a content less than 50%, the laterite corresponds to ferruginous soil; otherwise, it is known as ferralitic (Chatelin, 1969; Colleuille et al., 1994). Both lateritic soils are characterized mainly by the presence of quartz, goethite, hematite, kaolinite, and gibbsite (Maignien, 1966).

In West and Central Africa, laterite gravels are used as base and sub-base layers materials. They represent the fraction of soil with a 0/D grain size (i.e. 0/20 to 0/40 mm), with 10 to 35% fines passing through 80 µm sieve and 20 to 60% retained on 2 mm sieve (Bohi, 2008). According to the literature, the use of lateritic gravels depends on their geotechnical characteristics such as Atterberg limits, the fragmentability coefficient and the California Bearing Ratio (CBR) (Ndiaye et al., 2011). Submitted to a water exposure during lifespan or characterized by a low CBR index, lateritic gravels require a chemical treatment to enhance their mechanical and geotechnical characteristics before their employment as road materials. Most of the research dedicated to laterite treatment has focused on methodologies involving hydraulic binders, mainly cement, often hydraulic lime or both of them (Mengue et al., 2017; Qian et al., 2017). But the binders' addition raises several issues. The cement manufacturing is responsible for greenhouse gas emissions, with a ratio of 0.6 kg of carbon dioxide per kilogram of cement produced (Dahanni et al., 2023). Moreover, the cement introduction leads to rigid lateritic road layers, prone to cracking and less adapted to the passage of overloaded heavy-duty trucks in African context, which requires more flexible pavements.

In these conditions, bitumen emulsion appears as a good alternative to cement addition for lateritic soil treatment. Emulsion corresponds to a dispersion of bitumen fine droplets in an aqueous phase containing a surfactant, a molecule able to reduce the surface tension of water, thereby making the system more stable. Since the 1970s, South Africa has been a pioneer in the implementation and standardization of soil treatment with bitumen emulsion (Hitch and Russell, 1977; Collings et al., 2020). Now, the technique has aroused a real interest in the past fifteen years in many African countries. Empirical research conducted on laterite treated with bitumen emulsion has yielded favorable results (de Sabran-Pontevès, 2020), but the efficiency of the treatment may vary depending on the considered site and the various trends observed were not clearly interpreted until now. The extant literature on emulsion treated-laterite focuses primarily on mechanical performance, without systematic correlation with the composition and the process parameters (Matthew and Paul, 2018; Oluyemi-Ayibiowu, 2019). Nonetheless, the successful implementation of the treatment depends on several factors, such as the intrinsic properties of laterites, especially their mineralogy and geotechnical properties, as well as the characteristics of applied emulsions (surfactant nature and emulsion content). Furthermore, the interaction between lateritic particles and bitumen emulsion remains a determining factor in the effectiveness of the construction process.

To date, no investigations concerned the physico-chemical aspects of both laterite and bitumen emulsion. Most of studies on the emulsion/mineral substrate interface dealt with road aggregates and reclaimed asphalt pavements (Ziyani, 2013; Ziyani et al., 2014). However, these materials differ from laterites, characterized by a high iron and aluminium oxide content. The present paper aims to evaluate the behavior of laterites treated with bitumen emulsion based on their specific components. To address this objective, two emulsions and three laterites were selected for investigation, with a view to assessing their intrinsic physical, chemical, and mechanical characteristics.

2 MATERIALS AND METHODS

2.1 *Materials*

Two surfactants named A and B were selected for emulsion production. Molecule A is derived from fatty amides, while surfactant B is a combination of fatty acids and polyamines. The emulsifier content was set at 1%wt of the emulsion.

The examined bituminous binders were cationic emulsions comprising 60%wt of 50/70 penetration grade bitumen (C60) (Table 1). To achieve a pH value of 2, hydrochloric acid (HCl) was added to the aqueous phase.

After the emulsions' fabrication, a series of characterization tests were performed. The most relevant were the evaluation of the Forshammer breaking index, according to the standardized method NF EN 13075-1 (European Committee for Standardization, 2016a) and the determination of binder content, in accordance with NF EN 16849 (European Committee for Standardization, 2016b). Table 1 shows the results of the bitumen emulsions' properties. Emulsions A and B are classified as slow-setting, since their breaking value is greater than 170. In addition, the breaking index of emulsion B (360 ± 4) is slightly higher than that of A (334 ± 11), which means that the rupture of emulsion A occurs more rapidly compared to emulsion B.

Table 1. Bitumen emulsion characterization.

	A	B
Binder Content (%)	59.9 ± 0.1	60.1 ± 0.1
Forshammer Breaking Value	334 ± 11	360 ± 4

The emulsions were used to stabilize three laterites, named according to the nomenclature indicated in Table 2, and collected in Cameroon and Côte d'Ivoire. Characterized by a red color, they were extracted at depths ranging from 0 to 0.6 meter below the topsoil level. Laterites, as natural materials, were submitted to climatic and topographical conditions that cause differences in grain size and color depending on depth, due to the degree of laterization. After sub-sampling and homogenization, laterites were characterized before the treatment application using emulsion.

Table 2. Information on laterite samples.

Country	Location	Nomenclature	Gradation (0/D)
Cameroon	Sangmélima	CAM-SANGM	0/20
Côte d'Ivoire	Akoupé-Kotobi-Bongouanou road	CIV-RAKB	0/20
	Satama-Sokoura-Sandégué road	CIV-R3S	0/20

2.2 *Chemical and mineralogical characterization of raw laterites*

In this paper, the chemical characteristics of the laterite samples, represented by the R resultant, were determined to classify them into ferruginous and ferrallitic laterites. The R parameter designs the sum of iron (Fe_2O_3) and aluminum (Al_2O_3) oxide contents (Equation (1)). The lateritic gravel is considered as ferruginous if R<50%, otherwise it is classified as ferrallitic. Such classification is independent of the country origin of the samples.

$$R(\%) = \%Fe_2O_3 + \%Al_2O_3 \qquad (1)$$

To this end, the experiments were conducted on a representative 0/20 fraction. The samples were ground using a Retsch PM 400 planetary ball mill at 200 rpm for 20 minutes and then sieved to 63µm before testing. The chemical composition of the laterites was measured by X-ray fluorescence (XRF) method after pellets forming (10 g laterite was mixed with 1 g wax) using a Bruker S2 Ranger spectrometer equipped with an X-ray tube and a palladium anode.

The mineralogical composition of laterite was obtained by X-ray diffractometry (XRD). This test was performed via a Bruker D2 Phaser instrument, equipped with a copper anode (CuKα, λ = 1.54Å), operating at a voltage of 30 kV and a current of 10 mA. A 2θ scan was recorded from 5° to 70°, with a step size of 0.02° and a goniometer radius of 141 mm.

2.3 *Geotechnical characterization of raw laterites*

Figure 1 depicts the particle size distributions of laterites after dry sieving. The curves show significant differences in terms of particle size for sieves larger than 1 mm. These variations affect the cohesion of the compacted test specimens, which potentially influence the mechanical tests results.

The objective of this study is to evaluate the influence of laterite and emulsion nature on the mechanical behavior of treated laterites. For this purpose, the particle size distribution curve of CIV-R3S laterite was chosen as a granulometric reference curve (to be replicated on two other tested laterites). This choice allows to limit the influence of soil gradation and to favor the manufacture of more cohesive lateritic specimens, since CIV-R3S contains more fine particles (Figure 1). To perform the reconstruction of the particle size distribution curves of CAM-SANG and CIV-RAKB, the last two laterites were sieved in six granular fractions, notably 0/1, 1/2, 2/6.3, 6.3/12.5, 12.5/16, 16/20, and the contents of each granular fraction were chosen so as to obtain the particle size distribution curve of CIV-R3S laterite. Once this step was completed, all fractions were homogenized and ready for testing.

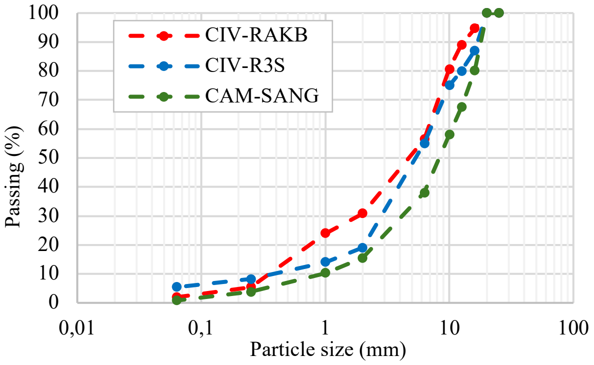

Figure 1. Particle size distribution.

The Atterberg limits have been measured according to NF EN ISO 17892-12 (European Committee for Standardization, 2018b) standard to analyze soil plasticity.

The Proctor compaction test was used to assess the maximum dry density and the optimum moisture content (OMC) for the samples' compaction. This OMC was determined using the modified Proctor test described in NF EN 13286-2 (European Committee for Standardization, 2010). The California bearing ratio test was performed according to NF EN 13286-47 (European Committee for Standardization, 2021) to evaluate the mechanical characteristics of raw laterites.

All these geotechnical properties allowed to classify the different laterites according to the "Guide de Terrassement Routier (GTR)", which is the most widely used in the West African context. The classification of laterite soils was also undertaken in accordance with both the American Association of State Highway and Transportation Officials (AASHTO) and the Unified Soil Classification System (USCS).

2.4 *Mechanical characterization of raw and bitumen emulsion-treated laterites*

The raw and treated laterites were formulated at a water content equal to the OMC given by modified Proctor test (2.3) and corresponding to the amount of water in the raw laterite plus the additional water. In the case of treated laterites, the required additional water was determined by considering the water contained in the emulsion. A manual blending protocol was applied for emulsion treatment. First, 7 kg of raw laterite was homogenized for 30 seconds, then water was introduced, and the mixture was stirred for 3 minutes, before incorporating the bitumen emulsion and mixing again for 3 minutes. The relatively small quantity of material ensured effective homogenization, which was assessed visually. Each laterite was treated with 3 and 5%wt emulsion (A and B) relative to the laterite weight (Figure 2). Then, each mixture was placed into six molds and compacted using the Marshall compaction method at 75 blows per face according to NF EN 12697-30 (European Committee for Standardization, 2018a). Then, the samples (101.1 mm in diameter and 62 ± 1 mm in height) were cured in a Weiss Pharma 500-L climatic chamber at 35°C and 80% relative humidity.

The mechanical properties of the treated and untreated laterites were assessed after 7 and 28 days of curing using stiffness modulus (E) and indirect tensile strength (ITS) tests. Experiments ascribed to stiffness modulus were carried out at temperatures of 15°C and 35°C, using standard NF EN 12697-26 – Annex C (European Committee for Standardization, 2022), whereas the indirect tensile strength tests were performed according to NF EN 13286-42 (European Committee for Standardization, 2003) at only 35 °C.

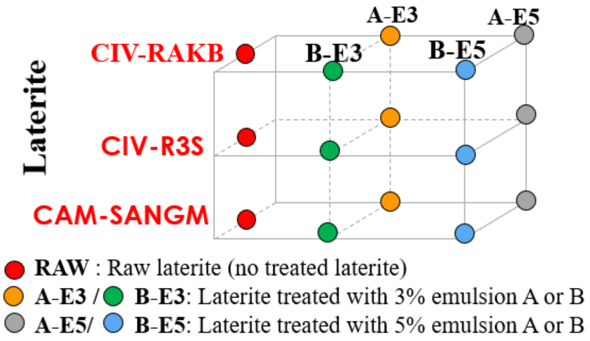

Figure 2. The different combinations of raw and treated laterites.

3 RESULTS AND DISCUSSION

3.1 *Lateritic gravels characteristics*

3.1.1 *Chemical and mineralogical characteristics*

The results of the X-ray fluorescence test are summarized in Table 3. As expected, iron, aluminum, and silicon oxides were the main species found in the laterites. The sum of these species exceeded 80%. Furthermore, CAM-SANG and CIV-RAKB laterites had similar chemical characteristics, with high iron oxide content (around 40%) and average aluminum and silicon oxide content (25%). However, CIV-R3S standed out due to its low iron and aluminum oxide content (28% and 15%, respectively) and high silicon oxide content (36%). These observations suggest that the laterites have undergone different laterisation processes, independent of their country origin. Table 3 also depicts the R resultant (Equation (1)) of the laterites. As indicated, the CIV-R3S laterite is ferruginous, while CIV-RAKB and CAM-SANG are ferrallitic.

Table 3. Chemical composition and classification of laterites. Class: classification, LOI: loss on ignition.

	CIV-RAKB	CIV-R3S	CAM-SANG
Fe_2O_3 (%)	37.3	28.4	41.7
Al_2O_3 (%)	26.5	15.1	22.0
SiO_2 (%)	24.2	36.2	23.4
LOI (%)	8.7	8.5	10.3
Others (%)	3.3	11.8	2.6
R (%) (%Al_2O_3+Fe_2O_3)	63.8	43.5	63.7
Class.	Ferrallitic	Ferruginous	Ferrallitic

Figure 3 represents the different mineral phases obtained by XRD in the studied laterites. The common mineral species found in samples were goethite, hematite, kaolinite, and quartz. Results agreed with the literature (Kumar et al., 2022) as well as previous measurement of chemical composition. The loss on ignition observed in Table 3 can be attributed to the dehydroxylation of goethite at 300°C and of kaolinite at 550°C (Koua-Koffi et al., 2018). Moreover, diffractogram analysis pointed out a similarity between the crystal structures of CIV-R3S and CIV-RAKB. However, the quartz peaks in CIV-R3S were more pronounced, while gibbsite was much more prevalent in CIV-RAKB.

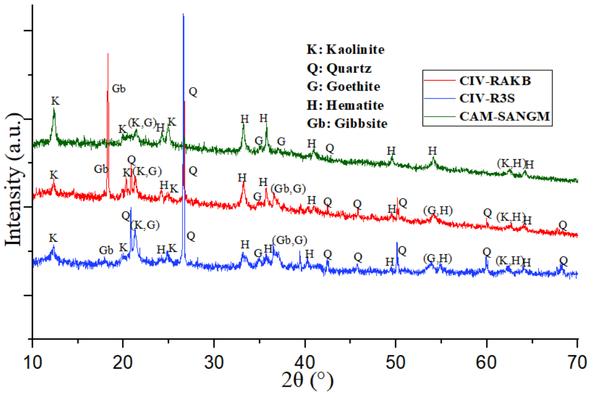

Figure 3. X-ray diffractograms of laterites.

3.1.2 *Geotechnical characteristics*

Table 4 summarizes the studied laterites geotechnical classifications, while Figure 4 illustrates the plasticity status of the tested soils, following the Casagrande texture diagram. Each classification (Table 4) confirmed that the tested materials were lateritic gravels. Additionally, the analysis of the Casagrande diagram indicated that the CIV-RAKB and CIV-R3S laterites had a clayey texture, while the CAM-SANG laterite showed silty characteristics (Table 4). A similarity appeared between the CBR indices and the optimum moisture content of CIV-RAKB and CIV-R3S. Furthermore, the CBR index of the three laterites was below 60, which means that laterites required imperatively a treatment before their use as base layer materials.

Table 4. Geotechnical characteristics of laterites. Class.: classification; SC: clayey sand; CL: clay low plasticity; MH: silt high plasticity

Evaluation	Evaluation type	CIV-RAKB	CIV-R3S	CAM-SANG
Class.	AASHTO class.	A-2-6	A-2-6	A-2-6
	GTR class.	I-2	I-2	I-2
	USCS class.	SC	SC	SC
Plasticity	Liquid Limit (%)	26.3	18.7	67.2
	Plasticity Index (%)	14.6 ± 1.7	12.3 ± 1.2	23.9 ± 1.3
	Casagrande class.	CL	CL	MH
Load-bearing	CBR Index	45.5 ± 2.1	46.5 ± 4.9	50.5 ± 4.9
Compaction	Optimum Moisture Content (%)	8.5 ± 1.9	8.5 ± 0.5	11.3 ± 0.5

3.2 *Treatment of lateritic gravels with bitumen emulsion*

Figure 4 shows stiffness modulus results for raw and treated laterites at 15 and 35°C, after a curing period of 7 and 28 days. Figure 5 displays indirect tensile strength values at 35°C, after a period of 7 and 28 days of curing.

3.2.1 *Influence of laterite nature*

Whatever the test temperature and curing time, raw CIV-R3S laterite had the highest stiffness modulus (E≈2700 MPa, Figure 4) and indirect tensile strength (ITS≈ 190 kPa, Figure 5), followed by that of raw CIV-RAKB (E≈1950 MPa, ITS≈ 190 kPa). On the contrary, raw CAM-SANG laterite had the weakest mechanical characteristics (E≈124 MPa, ITS≈ 130 kPa). The results for this sample can be explained by its ferrallitic and silty nature.

An evaluation of the characteristics of emulsion-treated laterites revealed that CIV-R3S stands out due to its excellent mechanical performance. This would mean that a ferruginous laterite offers better mechanical characteristics before and after treatment due to its clayey texture (Figure 4). Ferruginous soils are characterized by their high clay content, which confers excellent mechanical properties, surpassing even those of silty laterites. This phenomenon can be attributed to the intrinsic cohesion exhibited by the clay particles, which form a robust bond network (Yin et al., 2021).

3.2.2 *Influence of curing time and temperature*

Figures 5 and 6 display no significant changes in the mechanical characteristics of compacted raw laterites after 7- and 28-days curing. Conversely, an improvement in stiffness and indirect tensile strength was observed after a longer curing period when the laterites were treated with bitumen emulsion. Mass weighing showed that the density of the different formulations remained stable after 7 days of curing.

When emulsion and granular fractions are mixed, the emulsion adheres preferentially to the fine particles (sieved to 63µm) and coats the granular skeleton, because they have the largest specific surface area. This combination is called "mastic". The mastic stiffness increases as water evaporates (Bérubé, 2019; Al-Mohammedawi and Mollenhauer, 2020). So, the enhancement in the mechanical properties can be ascribed to the stiffening of the mastic, which is formed by the interaction between bitumen emulsion and lateritic fines.

Finally, a significant decrease in stiffness modulus between 15°C and 35°C was observed. Considering that the used bitumen softening point is set at 48°C, a viscous material is obtained at this temperature. Therefore, the mastic inherited the viscoelastic character of bitumen with a temperature dependency. The effect of temperature on the mastic properties can explain the loss of stiffness modulus at 35°C.

3.2.3 *Influence of bitumen emulsion*

The values of the stiffness modulus and indirect tensile strength in Figures 5 and 6 underlined a significant improvement in the mechanical characteristics after treatment with bitumen emulsion over a period of 28 days curing. In addition, results highlighted a difference in post-treatment behavior depending on the type of laterite. Indeed, the stiffness modulus of the CIV-R3S modulus increased by 45%, from 2,600 MPa at 7 days to 3,900 MPa after treatment. On the other hand, CAM-SANG laterite showed a gain in stiffness modulus of only 1,450% when treated with 3% emulsion. In all cases, the treatment with bitumen emulsion significantly improved the poor mechanical performance of laterites. Furthermore, increasing the emulsion content leads to an enhancement in the stiffness and tensile strength of laterites. This trend could be attributed to the bitumen film around the lateritic fines. In fact, when the emulsion and the laterite were mixed, a bitumen film formed around the finest particles and the thickness of the film influenced the granular interactions within the mastic formed (Robati, Carter and Perraton, 2015). So, the stiffness of the mastic appeared proportional to this thickness according to the literature. Increasing the emulsion content leads to an increase of the bitumen film thickness around the lateritic fine particles. As a result, the stiffness of the mixture increased.

A focus on Figure 4 and 6 also shows that the type of emulsion played an important role in the behavior of laterite treated with bitumen emulsion, even at a low bitumen emulsion dosage (i.e. 3%). At a constant bitumen emulsion content, emulsion B systematically offered higher strength than emulsion A when incorporated to CIV-R3S ferruginous laterite, and in most cases, when added to CIV-RAKB and CIV-R3S, some ferrallitic laterites. One exception was noted at 35°C and 28 days curing, for which ITS values exhibited higher mechanical characteristics using emulsion A for CAM-SANG and CIV-RAKB laterites.

To better understand the treated laterites' properties, when bitumen emulsion meets a granular surface, several physical and chemical phenomena occur at interfaces, such as the adsorption of surfactants. The more effective a surfactant is, the more it adsorbs during treatment (Günister et al., 2006; Liu et al., 2023).

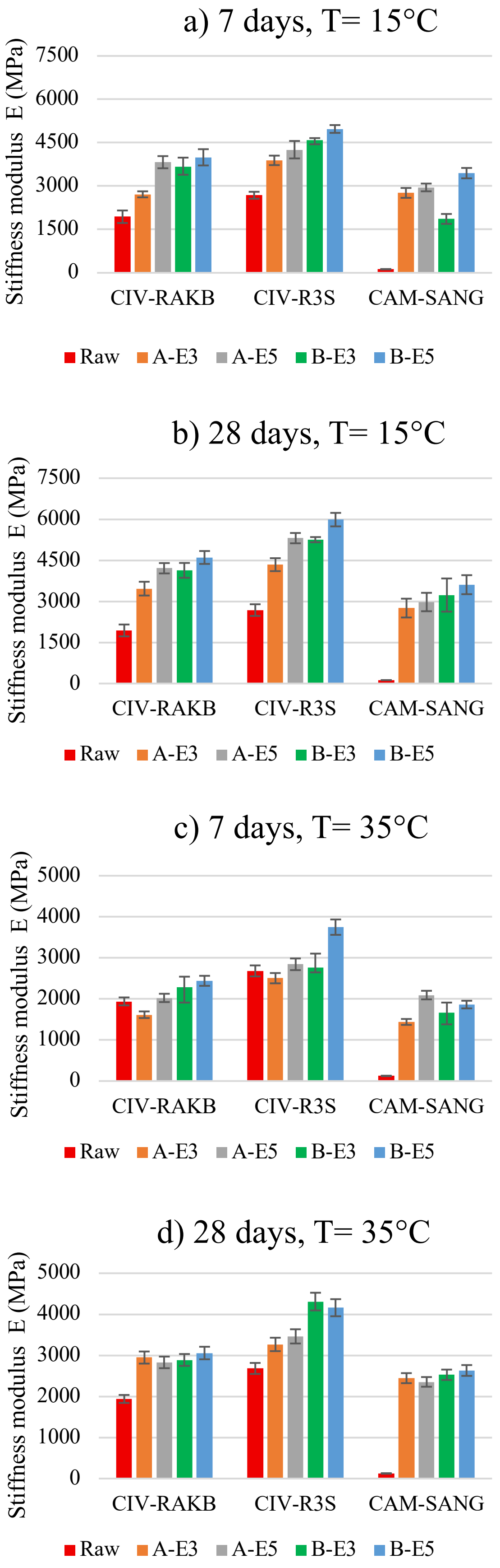

Figure 4. Stiffness modulus of raw and treated laterites, a) 15°C, 7 days curing, b) 15°C, 28 days curing, c) 35°C, 7 days curing and d) 35°C, 28 days curing.

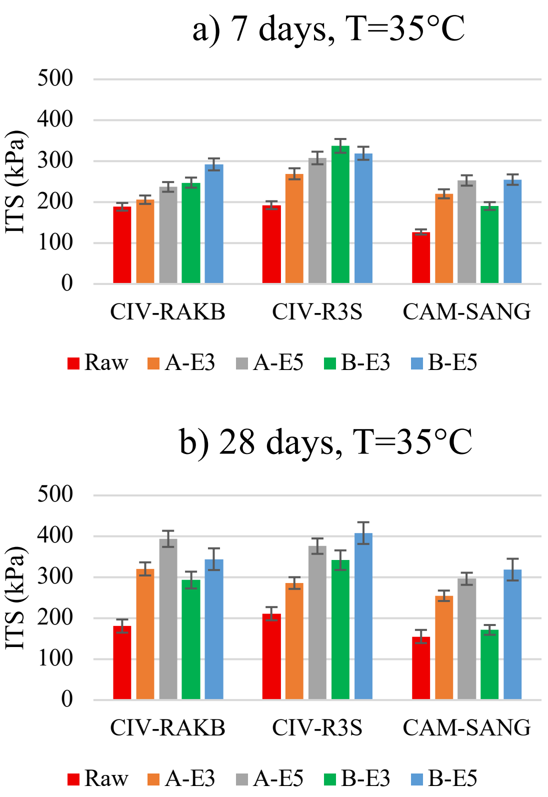

Figure 5. Indirect tensile strength at 35°C, a) 7 days curing and b) 28 days curing.

Based on this principle, the higher performance of laterites treated with emulsion B indicated a stronger affinity between lateritic fines and surfactant B. The presence of polyamines in such surfactant, which are absent in surfactant A, might be responsible for higher affinity between laterites and emulsion B. As a conclusion, the behavior of laterites treated with bitumen emulsion was influenced by both the nature of the surfactant as well as the laterite chemical composition.

4 CONCLUSIONS

Laterites used in pavements as base or sub-base courses usually require strengthening through a treatment with cement or hydrated lime, and alternatively bitumen emulsion providing lower harmful environmental impact. However, current knowledge of the latter technique remains limited and comes mainly from feedback from construction sites. Furthermore, the existing studies do not consider variables such as the nature of the laterite or the type of emulsion. This paper analyzed the impact of two emulsions on the mechanical behavior of three treated laterites. As a conclusion, the characteristics of laterites, influenced by their geological nature, impacted their mechanical performance: ferruginous laterites exhibited higher mechanical properties compared to ferrallitic ones. In addition, the application of an emulsion treatment, even at low dosage (3%wt emulsion), resulted in a significant improvement in the mechanical properties, which validated the suitability of the bitumen emulsion technique for laterite treatment. The stiffness modulus and indirect tensile strength of samples treated with bitumen emulsion also increased with the curing period, whereas the characteristics of natural laterites remained constant over time.

Considering the curing temperature effect, results underlined a loss of stiffness modulus with a marked decrease between 15°C and 35°C, which is of key importance in the

African context. Therefore, temperature appeared as a crucial factor for adequate design of laterite road treatment with bitumen emulsion.

The type of emulsion exerted a significant effect on the bitumen emulsion treatment of laterites, even at low concentrations. The treatment with emulsion B induced higher mechanical characteristics, compared to laterite treatment with surfactant A. However, a long-term affinity between emulsion A and ferrallitic laterites was observed in ITS tests. Conversely, surfactant B exhibited enhanced properties when it is employed to treat ferruginous laterites.

Finally, the results of this study introduced a novel approach to the use of laterite in road construction but to go deeper in the understanding of laterite behavior, the impact of water on the mechanical properties of treated laterites should be considered in further research. Indeed, the mechanical tests in this study were performed under dry conditions. Furthermore, questions remain regarding the design criteria for laterite pavements treated with bitumen emulsion (i.e. the impact of the surfactant content, fixed at 1% in this study, as well as the physico-chemical interactions at the surfactant-laterite interface).

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