



# Article Study of the Mining Waste in the Production of Calcined Aggregate for Use in Pavement

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**Abstract**: The aim of the present study was to evaluate the technical feasibility of using a calcined aggregate with mining residue in different pavement layers, including the base, subbase, and wearing course layers. For this purpose, physical characterizations of the residue and clay and the production of calcined aggregates at temperatures ranging from 800 °C to 1100 °C were performed. Additionally, the suitability levels of these aggregates in pavement layers were assessed, considering the present standards. The physical characterization results indicated that the studied clay was suitable for manufacturing calcined clay aggregates since the particle size distribution showed ceramic potential according to the Winkler diagram, and it presented a plasticity index (PI) higher than 15%. In the tests of boiling-induced mass loss and unit mass, the values obtained were within the limits established by the standards, being lower than 10% and 0.88 g/cm<sup>3</sup>, respectively. Regarding the abrasion loss test, the M1100 aggregate showed Los Angeles abrasion values lower than the limit established by the standard, demonstrating its potential as an artificial aggregate in pavement applications.

Keywords: pavement; aggregate; calcined clay



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# 1. Introduction

Brazil has witnessed two major catastrophes in the last decade due to the rupture of iron ore tailings containment dams. On 5 November 2015, the Fundão dam collapsed, resulting in the spillage of over 50 million cubic metres of mining waste into the Rio Doce [1]. Four years later, in 2019, the tailings dam of Córrego do Feijão, located in Brumadinho, Minas Gerais, collapsed. This tragedy led to the loss of over 250 human lives and the spilling of approximately 12 million cubic metres of ore tailings into the Paraopeba River and nearby areas [2].

These events caused a significant loss of human life, extensive environmental degradation, and social and economic losses, and were associated with mining activities that exert adverse effects on the ecosystem [3]. According to the National Mining Agency [4], there are 928 registered mining waste containment dams in Brazil, of which 252 have a high associated damage potential (DPA). This indicator is related to the potential damage caused by eventual dam rupture, and it is determined by the construction and conservation characteristics of the dams. Of the dams, 51 are classified as high-risk according to the categorical risk index (CRI).

Mining waste obtained at different stages of the mining process comprises materials with favourable granulometry (sandy tailings) from a geotechnical perspective and materials with unfavourable or deficient characteristics, such as fine-grained tailings, which are referred to as fine tailings or slimes [5].

Apaza et al. [6] evaluated the incorporation of sandy tailings composed of quartz minerals (88%) and haematite (9%) as aggregates from the iron-ore-refining process in cold asphalt microsurfacing. The results were satisfactory, and the iron ore waste did not present

any chemical, environmental, mineralogical, or physical restrictions that would affect its use as aggregates in microsurfacing mixtures.

Due to the intrinsic environmental risks associated with the mineral exploitation process, Rybak et al. [7,8] conducted an investigation based on the experience of the Russian mining industry. The scholars proposed a process capable of eliminating harmful accumulated waste, recovering the precious metals contained in these residues, and using the remaining waste as a landfill material in the form of an aggregate or binder. The experimental results suggested a potential use of landfill material waste, which could prevent disasters resulting from the collapse of abandoned galleries and protect the environment against toxic pollution.

Conversely, fine-grained tailings consisting of silt and clay fractions are typically plastic and highly compressible materials, as described by Lima [9]. These characteristics limit their potential for direct reuse. However, their utilization in the production of calcined aggregates for pavement is a promising alternative, as assessed by Sultan [10]. In this study, the results indicated that regardless of whether they were untreated or stabilized with cement or asphalt, mining wastes exhibited suitable engineering characteristics to be harnessed as alternative materials in pavement construction.

In this scenario, various approaches emerged that aimed at the reutilization of waste materials originating from mining. However, regarding their applications in road construction, the utilization of mine tailings has not yet been extensively explored. Additionally, there is a significant gap in the legislation specifically addressing this issue, as explained in Ref. [11].

In addition to the aforementioned alternatives, there is a concern regarding the use of industrial waste that is in line with the principles of waste-free mining [12,13]. Rybak et al. [3] confirmed that this phenomenon is feasible through the systematic and integrated use of mineral resources, improving the efficiency in the development of mineral deposits and the economic effects resulting from the prevention of environmental impacts.

Numerous scholars have evaluated the use of waste materials in engineering as a sustainable approach for aggregating production. An example is the work of Fan et al. [14], who investigated the use of ashes from municipal solid waste incineration (MSWIBA) and calcined clay as ecologically friendly artificial aggregates (EFAAs). With a compressive strength exceeding 30 MPa at 28 days, concrete produced using this artificial aggregate showed low toxicity, low energy consumption, and low CO<sub>2</sub> emissions, making it suitable for use in construction. However, the use of coal ash waste as a substitute for fine aggregates in lightweight concrete could reduce the strength when subjected to acidic environments, as observed in the work of Ghazali et al. [15]. Nevertheless, when properly thermally treated with an appropriate burning plan, the fluxing elements present in the waste could confer satisfactory strength to the aggregates, as concluded by Cabral et al. [16]. These authors obtained low-cost aggregates through the calcination of clayey soils, making them suitable for use as pavement materials.

Regarding calcined aggregates, the Brazilian Army (EB) was a pioneer in the production of artificial calcined clay aggregates in Brazil. The use of these materials in pavement construction became significantly important due to the challenges faced by the EB's engineering corps regarding the scarcity of rock deposits in the northern region of the country [17].

The topic was addressed by Batista [18], who evaluated the use of calcined clay aggregates in asphalt mixtures for pavement in the Amazon region. It was concluded that the results of the resilience modulus using calcined clay produced at a temperature of 900 °C for 30 min demonstrated the technical feasibility of using the aggregate in asphalt mixtures for pavement, differing from the values obtained for the region soil.

The results were corroborated by Silva et al. [19], who indicated that the application of sintered calcined clay aggregates (SACCs) in asphalt concrete mixtures for road pavement was a good alternative to natural granular aggregates in regions where these materials are scarce. The tests performed under cyclic loading indicated that the performance of SACC

in AC50/70 asphalt concrete mixtures was comparable to that of traditional aggregates. Additionally, the study showed that the use of SACC could reduce the carbon footprint of asphalt concrete production.

Santos [20] investigated the use of calcined clay aggregates as coarse aggregates in asphalt coatings, considering a firing temperature of 900 °C and firing time of 15 min. A comparison of the results for asphalt mixtures using rolled pebbles (commonly used in the Amazon region) and calcined clay aggregates showed that in certain aspects, such as permanent deformation, the mixture with calcined clay presented better results, better adhesion among its components, and consequently, higher mechanical resistance than the asphalt mixture containing pebbles. Moreover, increasing the firing temperature of the synthetic aggregates resulted in a reduction in asphalt cement consumption due to the transformation from the crystalline to the amorphous phase [21], indicating a strong possibility of using this aggregate as an alternative material for pavement in the region.

In addition to the surface layers, studies have been developed to assess the suitability of using calcined clay aggregates in other pavement layers. A noteworthy study conducted by Barbosa et al. [22] verified the viability of two soils in Acre: one to produce calcined clay aggregates, and another to produce soil–aggregate mixtures as the pavement base. The calcined aggregates were subjected to firing temperatures of 900 °C, 1000 °C, and 1100 °C, and all met the established limits of the present regulations for use as base materials for pavement. Regarding the addition of calcined aggregates to the soil–aggregate mixture, it was found that a proportion of 30% (by dry weight) was sufficient to promote the granulometric stabilization of the lateritic soil deposit used.

Cabral [23] proposed his own methodology for producing calcined clay, establishing parameters for the acceptance of the clay used by adjusting the final quality of the calcined aggregate. In summary, his methodology recommended the use of clays with a plasticity index (PI) higher than 15% and a granulometry preferably falling within regions B, C, or D of the Winkler diagram.

Other scholars have investigated the thermal behaviours of pavements composed of artificial aggregates. Yinfei et al. [24] studied the use of materials with phase change properties in asphalt pavements and how the granulation of lightweight aggregates (LWAs) could affect cooling performance. The results indicated that the granulation characteristics of LWAs significantly impacted the latent heat storage capacities of asphalt mixtures. Khan et al. [25] investigated the use of LWAs in asphalt mixtures and the base and subbase layers as a strategy for reducing frost damage in flexible pavements. The data indicated that the use of LWAs in asphalt mixtures and in the base and subbase layers could significantly reduce frost penetration in the subgrade and exhibit lower conductivity and diffusivity and higher specific thermal capacity values than conventional asphalt mixtures.

In other branches of the construction industry, scholars have indicated that the use of structural concrete with lightweight artificial aggregates could offer significant advantages in terms of weight reduction, improved thermal and acoustic insulation, and reduced energy consumption and  $CO_2$  emissions during production [26]. However, there are also some limitations and challenges associated with the use of these aggregates, such as the need for pretreatment of aggregates, reductions in mechanical strength and durability in certain cases, and the lack of standardization and regulations for the use of alternative materials.

As stated above, although there is a significant body of research on the use of mining waste to produce artificial aggregates for pavement, it is important to note that this subject requires further study. This phenomenon occurs due to the diverse nature of the waste characteristics, making accurate characterization essential for ensuring its proper application.

According to Segui et al. [11], utilizing mining waste as a construction resource offers dual advantages: the preservation of natural resources and the mitigation of the environmental repercussions of mining. However, the extensive implementation of mining waste in road construction is still in its infancy, largely due to a lack of comprehensive regulations. This gap arises from the multitude of exploited rock types, the heterogeneity of tailings, mining residues, and potential valuable byproducts earmarked for valorisation, alongside distinct environmental considerations.

Therefore, the aim of the present study is to evaluate the utilization of calcined aggregates produced from fine mining waste (sludge) as an alternative material for pavement construction that meets the criteria for selecting pavement materials established by the present standards in Brazil. The following hypothesis is presented in this study: the production of calcined aggregate from fine mining waste is a technically viable alternative in pavement construction.

# 2. Materials and Methods

The fine mining waste used in this research was obtained from the Samarco Mining Company, located in Mariana, Minas Gerais, Brazil. The clay was supplied by a ceramic company situated in the Taguará region, Rio de Janeiro, in Brazil.

The properties of the synthetic aggregate produced in this study were determined in three stages:

- First stage: physical, mechanical, and mineralogical characterization tests on the materials involved in synthetic aggregate production (pure clay, mining waste and clay/waste), such as Atterberg limits for liquidity (DNER-ME 122 [27]) and plasticity (NBR 7180 [28]), particle size analysis (DNER-ME 051 [29]), real density (DNER-ME 093 [30]), loss of mass after boiling (DNER-ME 225 [31]), expedited selection using the boiling method (DNER-ME 223 [32]), energy dispersive spectroscopy (EDS), and X-Ray Diffraction (XRD).
- Second stage: Aggregate production, with moulding carried out using an electric pug mill and calcination in a muffle-type furnace, with temperatures ranging from 800 °C to 1100 °C. The aggregates were divided into two sample groups: Group A represents aggregates produced from pure clay, and Group M represents aggregates produced from the clay/waste mixture (15% by weight of mining waste).
- Third stage: The following tests were conducted on calcined aggregates: unit weight (DNER-ME 152 [33]), loss of mass after boiling (DNER-ME 225 [31]), Los Angeles abrasion resistance (DNER-ME 222 [34]), shock loss of mass in the Treton apparatus (DNER-ME 399 [35]), and water absorption and density (DNER-ME 081 [36]).

#### Preparation of Samples

The extrusion process was chosen instead of using moulds for obtaining the samples, as this procedure has been employed in ceramic factories in Brazil.

The extrusion was carried out using an electric extruder with a hexagonal cross-section of 1 cm per side, and the moisture content for extrusion of both the pure clay and the mixture was 22%, which is close to the plasticity limit. The hexagonal-shaped nozzle was made of acrylic to ensure precision in the section dimensions through laser cutting and to provide a finished aggregate. After extrusion, the bar was cut into aggregates ranging from 5 mm to 15 mm in length, as shown in Figure 1.

As mentioned, two groups of samples were created: the A samples, consisting only of pure clay, and the M samples, referring to the clay–reject mixture, as shown in Figure 2. Subsequently, the aggregates were dried outdoors on metal trays for a period of seven days.

The calcination was carried out in a muffle furnace with four temperature stages—800 °C, 900 °C, 1000 °C, and 1100 °C—with a heating rate of 30 °C/min (adopted aiming at cost reduction in the manufacturing process), a dwell time of 30 min, and a furnace shutdown process for cooling.

XRD analyses were performed at CETEM (Mineral Technology Centre) using Bruker-D4 Endeavor equipment under the following operating conditions: Co K $\alpha$  radiation (40 kV/40 mA), goniometer speed of 0.02° 2 $\theta$  per step, counting time of 0.5 s per step, and data collection from 4 to 80° 2 $\theta$  using a LynxEye position-sensitive detector. Qualitative spectrum interpretations were made via comparison with patterns in the PDF02 database ICDD using Bruker DiffracPlus software.



Figure 1. Extrusion through an acrylic nozzle and cutting of the aggregates.



Figure 2. Samples produced in this study: (a) samples from Group A and (b) samples from Group M.

To assess the technical capability of using calcined clay–reject aggregate, the acceptance criteria recommended by the DNER-EM 230 [37] and DNER-ES 227 [38] standards were adopted. The synthetic aggregates of calcined clay must meet the conditions stipulated by the DNER-EM 230 [37] standard, as shown in Table 1.

Classification		Unit Weight		Loss of Mass after Boiling (%)	Los Angeles Abrasion (%)	
Class	Group	Max.	Min.	Max.	Max.	
	А	0.88	0.56	6	35	
Ι	В	0.88	0.56	6	40	
	С	0.88	0.56	10	45	
	А		0.88	6	35	
II	В		0.88	6	40	
	С		0.88	10	45	

Table 1. Classification of synthetic coarse aggregates according to DNER 230 [37].

The use of this aggregate in pavement was subjected to the class, group, and nature of the services, with the DNER-ES 227 [38] standard specifying this usage, as indicated in Table 2.

Nature of the Service	Class and Group
Surface treatments	IA
Asphalt concrete overlay	IA, IIA
Asphalt concrete base	IA, IB, IC, IIA, IIB, IIC
Exposed lightweight Portland cement concrete structures	IA
Portland cement concrete pavement	IA, AB
Portland cement concrete base	IA, IB, IC, IIA, IIB
Materials for flexible pavement	IA, IB, IC, IIA, IIB, IIC

Table 2. Synthetic coarse aggregate used according to DNER-ES 227 [38].

Impact resistance, as measured using the Treton apparatus (DNER-ME 399 [35]), is not a criterion specified in the DNER-ME 230 [37] standard for assessing the suitability of calcined clay artificial aggregates for pavement; however, since it was originally intended to evaluate stone materials, its inclusion was chosen in this study due to its direct relevance to traffic-induced stresses.

The acceptance criterion was set at a maximum impact loss value of 60% on the Treton apparatus, which was in line with the methodology proposed by Cabral [17], considering that Batista [18] and Cabral [23] adopted this limit in their research and obtained satisfactory results, even in experimental highway sections.

## 3. Results and Discussion

#### 3.1. Physical and Mineralogical Characterization

Based on the physical characterization results presented in Table 3, it can be observed that the studied residue is weakly plastic, indicating a PI < 7. It is worth noting that the fine iron ore residue from a mine in Yuhezhai, Yunnan, China, studied by Hu et al. [39], also exhibited a low plasticity of approximately 9%. Studies conducted by the Military Institute of Engineering indicate that to obtain a good-quality calcined clay, it is necessary for its particle size to fall within an acceptable range; furthermore, it should have high plasticity, or rather, a PI greater than 15%.

Test	Waste	Clay	Mixture
Liquid Limit (%)	20	39	20.5
Plastic Limit (%)	14	22	32.6
Plasticity Index (%)	6	17	12.1
Real Density (g/cm3)	3.93	2.6	2.8

Table 3. Physical characterization performed according to NBR 7180 [28].

Regarding the real density of  $3.93 \text{ g/cm}^3$ , the value is consistent with that of iron ore tailings and much higher than that of natural soils. This phenomenon was also observed in the study by Bastos [40], who found a density of  $3.55 \text{ g/cm}^3$ . The presence of haematite in the iron ore tailings justifies the high specific density of the material.

Concerning the particle size analysis, the tailings have high contents of fines (96% passing through a sieve with a size of  $n^{\circ}$  200).

The graphical illustration of the particle size distribution of the clay, mining waste, and mixture on the Winkler diagram, as proposed by Pracidelli and Menchiades [41], is displayed in Figure 3. It is evident that the mixture falls within the C region of the diagram, indicating its potential to yield a synthetic aggregate with satisfactory quality. However, the mixture is positioned at a critical point—on the borderline—and necessitates a high calcination temperature to attain the quality associated with the B range of the diagram.



**Figure 3.** Particle size distribution of the clay, mining waste and mixture represented on the Winkler diagram.

The EDS test on the clay reveals the predominant presence of silica, followed by aluminium, indicating that the clay used in the research is from the kaolinite group  $(Al_2Si_2O_5(OH)_4)$ . Regarding the result of the EDS test of the mining waste, a high concentration of iron is found, accounting for approximately 65% of the total.

After the characterization of the reject, an attempt is made to model the aggregate by extruding it using an electric pug mill. However, this attempt is unsuccessful as the equipment is clogged—possibly due to the low plasticity of the reject. Therefore, to increase the PI and enable the moulding of the aggregates, a mixture of clay and reject is used. The composition with 15% of this waste by mass is the maximum percentage that allows the use of the extruder.

In addition to the physical and chemical characterization tests of the clay and mining waste, a test recommended by the DNER-ME 223 [32] standard is conducted. This test assesses the potential of the material to produce calcined aggregate; it involves first moulding a cylindrical specimen measuring 4 cm in length and 1.5 cm in diameter and then calcining it at a temperature of 760 °C for 15 min. After calcination, the specimen undergoes boiling for 15 min in a pressure cooker.

At the end of the tests, it is observed that both clay and mining waste do not exhibit any visible change in volume or consistency, as seen in Figure 4. This finding indicates that both materials have the potential to be used in the production of calcined clay synthetic aggregates.



Figure 4. (a) Samples before the boiling test. (b) Samples after the boiling test.

Through XRD analyses, it is possible to identify the mineralogy of the clay, the mining waste, and the mixture analysed in this study (Table 4). The test shows that the studied clay consists of a clay mineral from the kaolinite group, as indicated by the EDS analysis.

The results also identify that the iron present in the mining waste is in the form of Goethite (iron oxide).

**Table 4.** Mineralogical compositions of the clay, waste, and mixture determined through X-Ray Diffraction (XRD).

Sample	Present Minerals
Soil	Kaolinite Orthoclase Halloysite Chromium Chlorite Quartz
Waste	Quartz Kaolinite Haematite Goethite
Mixture	Quartz Kaolinite Muscovite Microcline

## 3.2. Characterization of the Produced Synthetic Aggregates

## 3.2.1. Water Absorption Test

As seen in the graph illustrated in Figure 5, there is a slight reduction in water absorption with increasing temperature, indicating an increase in the amount of liquid phase resulting from the reaction of fluxing elements.



Figure 5. Water absorption of the aggregates.

According to the methodology to produce calcined clay proposed by Cabral [17], the water absorption of the aggregate cannot exceed 18%, which makes the use of calcined aggregate from both Group A and Group M unfeasible at the burning temperature of 800 °C. Therefore, the tests for bulk density, abrasion resistance, and real and bulk density are not conducted at the burning temperature of 800 °C.

#### 3.2.2. Loss Mass Test in the Treton Apparatus

The results of the tests for mass loss on the Treton apparatus (Figure 6) are satisfactory for all burning temperatures in both groups, as the mass loss does not exceed the limit of 60% established by Cabral [17].

It is observed in both loss mass tests that the higher the calcination temperature is, the lower the mass loss. At high calcination temperatures, the burning of volatile materials is increasingly complete. Therefore, it is observed that the mass loss during the boiling test



is relatively low at high temperatures due to the high conversion of volatile materials to gases and water vapour during combustion.

Figure 6. Loss of mass due to shock in the Treton apparatus.

# 3.2.3. Boiling Mass Loss Test

As shown in Figure 7, the results are adequate for all temperatures in both groups, being lower than 10%, as established by the DNER-ME 225 standard [31].



Figure 7. Mass loss due to boiling.

Furthermore, according to the graph illustrated below, there is an apparent plateau of mass loss after boiling between temperatures of 900 °C and 1000 °C, indicating a similar behaviour trend to the curves found by Cabral [17].

According to Ledbetter et al. [42], agitation in water disperses the materials, causing some abrasion loss. This abrasion related to Ref. [42] can be observed in Figure 8.



(a)

(b)

## 3.2.4. Mass Unit Test

The mass unit for Group M has higher values than Group A; this phenomenon is expected due to the elevated concentration of iron in the mining wastes, as illustrated in Figure 9.



Figure 9. Mass units.

All samples obtain values within the limit established by the DNER-ME 230 [37] standard (mass unit >  $0.88 \text{ g/cm}^3$ ). However, as Ledbetter et al. [42] observed in the field, during the execution of surface treatment with synthetic aggregate, LWAs degrade relatively fast in the wearing course layers. In this regard, the use of a dense aggregate, such as the synthetic aggregates obtained from the clay–reject mixture, may mitigate this reported issue.

## 3.2.5. Los Angeles Abrasion Test

Regarding the abrasion test, the Los Angeles abrasion values exceed the maximum limit of 45%, which was established by the DNER ME 230 [37] standard, in all samples, except for the calcined clay–reject aggregate sample with a burning temperature of 1100 °C (M1100), as shown in the graph illustrated in Figure 10. Therefore, since the test replicates the material service conditions, based on this parameter, only the M1100 sample demonstrates suitable resistance for use in pavement applications.



Figure 10. Los Angeles abrasion wear.

When associating the results with the XRD analyses, it is noted that the increase in calcination temperature and the emergence of the element in the mullite phase increases the resistance to wear for the aggregate.

After the Los Angeles abrasion test, the aggregates exhibit cubic visual shapes rather than lamellar shapes, as shown in Figure 11.

Comparing the results obtained by the scholars [43,44] that evaluated the use of phosphate ore waste, according to the Los Angeles abrasion limit used by the Egyptian

standard test methods for road construction, the M1100, M1000 and A1100 samples are satisfactory (<50%). However, relative to the limits used in Ref. [45], which involves the assessment of phosphate ore waste and references the standard of the Association Française de Normalization [46] (45% < Los Angeles abrasion < 58%), all samples meet the abrasion criterion.





## 3.2.6. Real and Bulk Density

The results of the real and bulk density test for coarse aggregate, according to the DNER-ME 081 standard [36], are presented in Table 5 and depicted in Figure 12.

Table 5. Results of the real and bulk density tests.

Sample	Real Density	Bulk Density
A900	2.30	1.70
A1000	2.23	1.93
A1100	2.24	1.93
M900	2.45	1.77
M1000	2.39	1.87
M1100	2.42	2.10



Figure 12. Real and bulk densities of Groups A and M.

As expected, since the waste originates from iron ore mining, the real densities of the aggregates in Group M are higher than those in Group A.

## 3.2.7. X-ray Diffraction (XRD)

The XRD analyses qualitatively show that the clay used in the clay–reject mixture is in the kaolinite group, confirming the results obtained via EDS. The XRD analyses also allow us to verify that the increase in temperature leads to changes in the chemical composition of the clay, while the reject maintains its initial chemical composition. Detailed results for the analysed groups are presented in Table 6.

Group	Sample	<b>Present Minerals</b>		Crystalline Systems
A	A800	Quartz Haematite Halloysite Goethite Albite	$\begin{array}{c} \mathrm{SiO}_2\\ \mathrm{Fe}_2\mathrm{O}_3\\ \mathrm{OH}_8\mathrm{Al}_2\mathrm{Si}_2\mathrm{O}_3\\ \mathrm{FeO(OH)}\\ \mathrm{NaAlSi}_3\mathrm{O}_8\end{array}$	Hexagonal Rhombohedral H. Axes Monoclinic Orthorhombic Triclinic
	A900	Quartz Haematite Halloysite Orthoclase	$\begin{array}{c} \mathrm{SiO}_2\\\mathrm{Fe_2O_3}\\\mathrm{OH_4Al_2Si_2O_5}\\\mathrm{K_{0.58}NaO_{0.42}AlSi_3O_8}\end{array}$	Hexagonal Rhombohedral H. Axes Hexagonal Triclinic
	A1000	Quartz Orthoclase	SiO <sub>2</sub> KAlSi <sub>3</sub> O <sub>8</sub>	Hexagonal Monoclinic
	A1100	Quartz Mullite	$\frac{\text{SiO}_2}{\text{Al}(\text{Al}_{69}\text{Si}_{1220})}$	-
M	M800	Quartz Hornblende Halloysite Microcline	SiO <sub>2</sub> (Na,K) <sub>0.72</sub> (Ca,Fe) <sub>2</sub> (Mg,Fe,Al) <sub>5</sub> (Si,Al) <sub>8</sub> O <sub>22</sub> OH <sub>2</sub> OH <sub>8</sub> Al <sub>2</sub> Si <sub>2</sub> O <sub>3</sub> (K <sub>0.95</sub> Na <sub>0.05</sub> )AlSi <sub>3</sub> O <sub>8</sub>	Hexagonal Monoclinic Hexagonal Triclinic
	M900	Quartz Hornblende Halloysite Orthoclase	$\begin{array}{c} {\rm SiO_2} \\ ({\rm Na},\!{\rm K})_{0.72}({\rm Ca},\!{\rm Fe})_2 \; ({\rm Mg},\!{\rm Fe},\!{\rm Al})_5({\rm Si},\!{\rm Al})_8 \; {\rm O}_{22} \; {\rm OH_2} \\ {\rm OH_8}{\rm Al_2}{\rm Si_2}{\rm O_3} \\ {\rm K}_{0.59}{\rm Ba}_{0.19}{\rm Na}_{0.22}({\rm Al}_{1.18}{\rm Si}_{2\cdot82}{\rm O}_8) \end{array}$	Hexagonal Monoclinic Monoclinic Monoclinic
	M1000	Quartz Hornblende Haematite Orthoclase	$\begin{array}{r} & SiO_2 \\ (Na,K)_{0.72}(Ca,Fe)_2 \ (Mg,Fe,Al)_5(Si,Al)_8O_{22} \ OH_2 \\ & Fe_2O_3 \\ K_{0.59}Ba_{0.19}Na_{0.22}(Al_{1.18}Si_{2.82}O_8) \end{array}$	Hexagonal Monoclinic Rhombohedral H. Axes Monoclinic
	M1100	Quartz Mullite	SiO <sub>2</sub> Al(Al <sub>69</sub> Si <sub>1220</sub> )	-

Table 6. Mineralogical characterization of the soil.

The feldspar present in the clay as Orthoclase and Albite plays a crucial role by acting as flux during the calcination process, reducing the material's porosity [47].

According to Santos [48], kaolinite, which is also present in clay, undergoes transformations in its chemical composition when exposed to temperatures; between 500 °C and 900 °C, it transforms into metakaolinite, and between 900 °C and 1550 °C, it transforms into mullite.

Hornblende, which is present in the calcined aggregates of Group M, is one of the main minerals of the amphibole group.

## 3.3. Analysis of the Technical Feasibility of Using the Synthetic Aggregate Produced

To evaluate the technical feasibility of utilizing aggregates made from calcined clay and reject materials, the criteria for acceptance proposed by the DNER-EM 230 [37] and DNER-ES 227 [38] guidelines are employed.

Group	Sample	Absorption	Treton Shock Loss	Loss of Mass after Boiling		Unit Weight (g/cm <sup>3</sup> )		Los Angeles Abrasion	
				Res.	Standard	Res.	Standard	Res.	Standard
А	A800	18.81%	50.3%	3.21%	<10%	-	-	-	-
	A900	16.21%	43.44%	2.52%	<10%	0.99	> 0.88	55.66%	<45%
	A1000	11.68%	39.91%	2.38%	<10%	1.03	> 0.88	54.11%	<45%
	A1100	9.28%	34.71%	1.1%	<10%	1.07	> 0.88	47.66%	<45%
М	M800	18.46%	51.93%	6.16%	<10%	-		-	-
	M900	17.04%	45.91%	2.9%	<10%	1.02	> 0.88	54.4%	<45%
	M1000	12.86%	39.81%	2.61%	<10%	1.10	> 0.88	50.12%	<45%
	M1100	7.68%	31.67%	0.82%	<10%	1.16	> 0.88	41.63%	<45%

 Table 7. Results of the tests conducted on the calcined aggregates.

mass loss after boiling, bulk density, and Los Angeles abrasion tests.

The water absorption results and shock loss tests using the Treton apparatus, relative to the limit values expressed in the methodology proposed by Cabral [17], obtain satisfactory results for firing temperatures starting at 900 °C (<60%).

In summary, Table 7 presents the results of the absorption, mass loss by Treton impact,

It is evident that the increase in temperature contributes to the reduction in water absorption in the aggregates due to the sintering of the fluxing materials present in the clay.

All samples of calcined aggregates produced in this study yield satisfactory results in the boiling loss of mass test, as each of them exhibit values below 10%, which is the maximum allowable limit for clay-type aggregates intended for use in pavement that is in accordance with the DNER-ES 227/89 [38] standard.

According to the standard DNER-EM 230 [37] for coarse synthetic aggregates of calcined clay, all the results fit within the established limits, except for the abrasion loss.

Only sample M1100 shows a result below the maximum limit set in the standard (41.6% < 45%). Furthermore, based on the values obtained from the tests of bulk density, loss of mass after boiling, and Los Angeles abrasion, according to the DNER-EM 230 [37] standard, the M1100 sample is classified as Class II and Group C. The material can be utilized by following the criteria established in the DNER-ES 227 [38] standard for an asphalt concrete base or flexible pavement base.

# 4. Conclusions

After conducting the tests in accordance with the Brazilian standard DNER-ME 230 [37], it was possible to identify the technical feasibility of using ore waste in the production of synthetic aggregates for pavement applications. The calcined aggregate produced using 100% clay exhibited unsatisfactory performance in the Los Angeles abrasion test. However, by incorporating mining waste, it became possible to transform a clay deposit that was previously unsuitable for producing a viable material from calcined clay aggregate.

The calcined aggregate obtained from the M1100 mixture with 15% waste by weight and a calcination temperature of 1100 °C demonstrated mechanical characteristics that were compatible with the requirements for use in asphalt concrete base and flexible pavement base materials, as defined by the DNER-ME standard.

The exclusive utilization of waste material in an electric pug mill for aggregate production proved unfeasible, likely due to the low plasticity of the waste, hindering its conformation during extrusion. However, mining waste showed potential when combined with clays to produce synthetic calcined aggregates, resulting in improvements in Los Angeles abrasion values relative to pure clay. The utilization of ore tailings in the production of calcined aggregates for pavement construction could not only enhance mechanical performance but also contribute to sustainable practices by mitigating environmental impacts.

Suggestions for Future Work and Recommendations of Research

- Evaluate the environmental aspects of using these aggregates in pavement layers.
- Assess potential environmental impacts due to the eventual release of toxic components resulting from material degradation over time.
- Study the expansion of the aggregate before conducting any experiments.

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# References

- Queiroz, H.M.; Nóbrega, G.N.; Ferreira, T.O.; Almeida, L.S.; Romero, T.B.; Santaella, S.T.; Bernardino, A.F.; Otero, X.L. The Samarco mine tailing disaster: A possible time-bomb for heavy metals contamination? *Sci. Total Environ.* 2018, 637, 498–506. [CrossRef] [PubMed]
- Thompson, F.; de Oliveira, B.C.; Cordeiro, M.C.; Masi, B.P.; Rangel, T.P.; Paz, P.; Freitas, T.; Lopes, G.; Silva, B.S.; Cabral, A.S. Severe impacts of the Brumadinho dam failure (Minas Gerais, Brazil) on the water quality of the Paraopeba River. *Sci. Total Environ.* 2020, 705, 135914. [CrossRef] [PubMed]
- Rybak, J.; Gorbatyuk, S.M.; Bujanovna-Syuryun, K.C.; Khairutdinov, A.M.; Tyulyaeva, Y.S.; Makarov, P.S. Utilization of Mineral Waste: A Method for Expanding the Mineral Resource Base of a Mining and Smelting Company. *Metallurgist* 2021, 64, 851–861. [CrossRef]
- 4. National Mining Agency—ANM. National Dam Safety Information System. Available online: https://www.gov.br/anm/pt-br (accessed on 30 May 2023). (In Portuguese)
- Porto, T.B.; Torres, A.C.A.; Gomes, R.C. Behavior of reinjectable and prestressed anchors in soil masses: Construction case study in Congonhas—Brazil (Case Study). Soil Rocks 2017, 40, 177–186. [CrossRef]
- 6. Apaza, F.R.; Guimarães, A.C.R.; Vivoni, A.M.; Schroder, R. Evaluation of the performance of iron ore waste as potential recycled aggregate for micro-surfacing type cold asphalt mixtures. *Constr. Build. Mater.* **2021**, *266*, 121020. [CrossRef]
- Rybak, J.; Kongar-Syuryun, C.; Tyulyaeva, Y.; Khayrutdinov, A.M. Creation of Backfill Materials Based on Industrial Waste. *Minerals* 2021, 11, 739. [CrossRef]
- 8. Rybak, J.; Adigamov, A.; Kongar-Syuryun, C.; Khayrutdinov, M.; Tyulyaeva, Y. Renewable-Resource Technologies in Mining and Metallurgical Enterprises Providing Environmental Safety. *Minerals* **2021**, *11*, 1145. [CrossRef]
- Lima, L.M.K. Retroanalysis of the Formation of An Aerial Method-Constructed Fine Mining Waste Deposit. Master's Dissertation, Federal University of Ouro Preto, Ouro Preto, MG, Brazil, 2006. Available online: http://www.repositorio.ufop.br/handle/1234 56789/2246 (accessed on 15 May 2023). (In Portuguese).
- 10. Sultan, H.A. Stabilized Copper Mill Tailings for Highway Construction. Transp. Res. Rec. 1979, 734, 15–19.
- 11. Segui, P.; Safhi, A.e.M.; Amrani, M.; Benzaazoua, M. Mining Wastes as Road Construction Material: A Review. *Minerals* **2023**, *13*, 90. [CrossRef]
- 12. Wellmer, F.W.; Becker-Platen, J. Sustainable development and the exploitation of mineral and energy resources: A review. *Int. J. Earth Sci.* 2002, *91*, 723–745. [CrossRef]
- 13. Phillips, J. Evaluating the level and nature of sustainable development of a mining operation: A new approach using the ideas of coupled environment-human systems. *Int. J. Min. Miner. Eng.* **2010**, *2*, 215–238. [CrossRef]
- Fan, X.; Li, Z.; Zhang, W.; Jin, H.; Liu, J.; Xing, F.; Tang, L. New applications of municipal solid waste incineration bottom ash (MSWIBA) and calcined clay in construction: Preparation and use of an eco-friendly artificial aggregate. *Constr. Build. Mater.* 2023, 387, 131629. [CrossRef]
- 15. Ghazali, N.; Muthusamy, K.; Abdullah, N.A.; Asri, M.I.M.; Jamaludin, N.F.A. Effect of coal bottom ash as partial sand replacement for lightweight aggregate concrete. *Key Eng. Mater.* **2022**, *912*, 119–125. [CrossRef]

- Cabral, E.M.; Sá, R.J.; Vieira, R.K.; Vasconcelos, R.P. Utilization of ceramic masses in the production of synthetic calcined clay aggregate for use in concrete. *Ceramics* 2008, 54, 404–410. (In Portuguese) [CrossRef]
- Cabral, G.L.L. Utilization of Calcined Clay Artificial Aggregate in Pavement Construction and Technology Enhancement. Ph.D. Thesis, Federal University of Rio de Janeiro, Rio de Janeiro, Brazil, 2011. Available online: http://www.coc.ufrj.br/pt/teses-dedoutorado/155-2011/1251-gustavo-da-luz-lima-cabral (accessed on 13 June 2023). (In Portuguese)
- Batista, F.G.S. Physical and Mechanistic Characterization of Calcined Clay Aggregates Produced from Fine Soils of BR-163/PA. Master's Thesis, Military Institute of Engineering, Rio de Janeiro, Brazil, 2004. (In Portuguese)
- 19. Silva, C.L.; Frota, H.O.; Frota, C.A. Sintered calcined clay as an alternative coarse aggregate for asphalt pavement construction. *Open J. Civ. Eng.* **2015**, *5*, 281–288. [CrossRef]
- Santos, M.G.R. Study of Synthetic Calcinated Clay Aggregates Behavior for Use in Pavement Asphalt for Manaus. Master's Thesis, University of Brasília, Brasília, Brazil, 2007. Available online: https://repositorio.unb.br/handle/10482/2919 (accessed on 25 June 2023). (In Portuguese)
- Campelo, N.S.; Campos, A.M.L.S.; Aragão, A.F. Comparative analysis of asphalt concrete mixtures employing pebbles and synthetic coarse aggregate of calcined clay in the Amazon region. *Int. J. Pavement Eng.* 2019, 20, 507–518. [CrossRef]
- Barbosa, V.H.R.; Marques, M.E.S.; Guimarães, A.C.R.; Silveira, V.L. Study of two soils from Acre to produce calcined clay aggregates and mixtures for pavement bases. In Proceedings of the 32nd Congress of Research and Teaching in Transportation of ANPET, Gramado, Brazil, 10–14 November 2018. (In Portuguese)
- 23. Cabral, G.L.L. Production methodology and use of calcined clay aggregates for pavement. Master's Thesis, Military Institute of Engineering, Rio de Janeiro, Brazil, 2005. (In Portuguese)
- 24. Yinfei, D.; Pusheng, L.; Jiacheng, W.; Hancheng, D.; Hao, W.; Yingtao, L. Effect of lightweight aggregate gradation on latent heat storage capacity of asphalt mixture for cooling asphalt pavement. *Constr. Build. Mater.* **2020**, 250, 118849. [CrossRef]
- 25. Khan, A.; Mrawira, D.; Hildebrand, E.E. Use of lightweight aggregate to mitigate frost damage in flexible pavements. *Int. J. Pavement Eng.* **2009**, *10*, 329–339. [CrossRef]
- 26. Agrawal, Y.; Gupta, T.; Sharma, R.; Panwar, N.L.; Siddique, S. A comprehensive review on the performance of structural lightweight aggregate concrete for sustainable construction. *Constr. Mater.* **2021**, *1*, 39–62. [CrossRef]
- 27. ME 122/94. Soils—Determination of Liquid Limit—Reference Method and Expedited Method. Department of National Roads and Highways—DNIT: Rio de Janeiro, Brazil, 1994. (In Portuguese)
- 28. NBR 7180/16. Soil—Determination of Plasticity Limit. Brazilian Association of Technical Standards—ABNT: Rio de Janeiro, Brazil, 2016. (In Portuguese)
- 29. ME 051/94. Soils—Particle Size Analysis. Department of National Roads and Highways—DNIT: Rio de Janeiro, Brazil, 1994. (In Portuguese)
- ME 093/94. Soils—Determination of Real Density. Department of National Roads and Highways—DNIT: Rio de Janeiro, Brazil, 1994. (In Portuguese)
- ME 225/94. Synthetic Aggregate of Calcined Clay—Determination of Mass Loss after Boiling. Department of National Roads and Highways—DNIT: Rio de Janeiro, Brazil, 1994. (In Portuguese)
- ME 223/94. Clays for the Production of Clay Synthetic Aggregate—Expedited Selection through the Boilling Process. Department of National Roads and Highways—DNIT: Rio de Janeiro, Brazil, 1994. (In Portuguese)
- ME 152/95. Aggregates in Loose State—Determination of Bulk Density. Department of National Roads and Highways—DNIT: Rio de Janeiro, Brazil, 1995. (In Portuguese)
- 34. ME 222/94. Synthetic Aggregate Manufactured with Clay—Abrasion Test. Department of National Roads and Highways—DNIT: Rio de Janeiro, Brazil, 1994. (In Portuguese)
- 35. ME 399/99. Aggregates—Determination of Shock Loss Using the Treton Apparatus. Department of National Roads and Highways—DNIT: Rio de Janeiro, Brazil, 1999. (In Portuguese)
- ME 081/98. Aggregates—Determination of Absorption and Bulk Density of Coarse Aggregate. Department of National Roads and Highways—DNIT: Rio de Janeiro, Brazil, 1998. (In Portuguese)
- EM 230/94. Coarse Synthetic Aggregates of Calcined Clay. Department of National Roads and Highways—DNIT: Rio de Janeiro, Brazil, 1994. (In Portuguese)
- ES 227/89. Coarse Synthetic Aggregates of Calcined Clay—Use in Road Works. National Department of Highways and Roads—DNER: Rio de Janeiro, Brazil, 1989.
- Hu, L.; Wu, H.; Zhang, L.; Zhang, P.; Wen, Q. Geotechnical Properties of Mine Tailings. J. Mater. Civ. Eng. 2017, 29, 04016220. [CrossRef]
- 40. Bastos, L.A.C.; Silva, G.C.; Mendes, J.C.; Peixoto, R.A.F. Using Iron Ore Tailings from Tailing Dams as Road Material. *J. Mater. Civ.* Eng. 2016, 28, 04016102. [CrossRef]
- 41. Pracidelli, S.; Melchiades, F.G. Importance of the granulometric composition of clay masses for red ceramics. *Ind. Ceram.* **1997**, *2*, 31–35. (In Portuguese)
- 42. Ledbetter, W.B.; Moore, W.M.; Gallaway, B.M. A Synthetic Coarse Aggregate Classification System-Final Report; Research Report No 81-15. Synthetic Aggregate Research. Study 2-8-65-81; Texas Transportation Institute: Arlington, TX, USA, 1971.
- 43. Ahmed, A.A.; Abouzeid, A.Z.M. Potential use of phosphate wastes as aggregates in road construction. *J. Eng. Sci.* 2009, 37, 413–422. [CrossRef]

- 44. Ahmed, A.A.M.; Abdel Kareem, K.H.; Altohamy, A.M.; Rizk, S.A.M. Potential Use of Mines and Quarries SolidWaste in Road Construction and as Replacement Soil Under Foundations. *J. Eng. Sci.* **2014**, *42*, 1094–1105. [CrossRef]
- Amrani, M.; Taha, Y.; Kchikach, A.; Benzaazoua, M.; Hakkou, R. Valorization of Phosphate MineWaste Rocks as Materials for Road Construction. *Minerals* 2019, 9, 237. [CrossRef]
- 46. *P18-573*; Aggregates. Los Angeles Test—Granulate. Los Angeles Pruefung; Association Française de Normalisation: Paris, France, 1990.
- 47. Baucia, J.A., Jr.; Koshimizu, L.; Gibertoni, C.; Morelli, M.R. Study of alternative fluxes for use in porcelain formulations. *Ceramics* **2010**, *56*, 262–272. (In Portuguese) [CrossRef]
- 48. Santos, H.S.; Kiyohara, P.; Coelho, A.C.V.; Santos, P.S. Study by electron microscopy of transformations during firing of highly alumina Brazilian clays. *Ceramics* 2006, *52*, 125–137. [CrossRef]

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