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Utilization of ferronickel slag in hot mix asphalt

Abstract

The growing concern in minimizing the disorderly disposal of waste in nature has been influencing measures that seek to give new environmentally sustainable and economically viable purposes for these materials. The use of steel slag aggregate in road paving emerges as an alternative for reducing the storage of this material in industrial yards, as well as contributing to a significant reduction in the cost of building a flexible road pavement. This study aimed to verify the technical feasibility of using ferronickel slag as an aggregate in the composition of hot mix asphalt. To this end, physical, chemical, mineralogical and environmental characterization tests of ferronickel slag were performed. The asphalt mixtures were dosed in accordance with the Marshall methodology, using the DNIT Range C with the use of ferronickel slag in the granulometric portions corresponding to the coarse aggregate, fine and filler, and the petroleum asphalt cement (PAC) 50/70. Based on the results, it can be stated that ferronickel slag demonstrates technical feasibility to be used as an aggregate in the composition of hot mix asphalt, meeting the requirements established by Brazilian standardization. In addition, it is an excellent environmental alternative because it uses a material previously treated as an environmental liability, avoiding the exploration of new natural deposits of stone aggregates.

Keywords: ferronickel slag; alternative aggregate; hot mix asphalt; asphalt paving; Marshall mix design.

1. Introduction

Brazil is a country of continental dimensions, and has as its main transportation modal, the road system that is responsible for more than 61% of the cargo transportation matrix and 95% of the passenger transportation. The transportation infrastructure has a fundamental function in the process of economic development of a nation, being able to influence even its productivity and foreign relationships through the activities of imports and exports that are directly linked to the situation of the transportation infrastructure of the country.

The Brazilian road network is

composed of 1,720,700.30 km of highways, of which only 213,452.80 km are paved, representing 12.4% of the total extension. The expansion of the paved highway network also does not keep pace with the growth of the vehicle fleet (CNT, 2019).

The engineering techniques cur-

rently used in road projects, as well as the methods applied in the sizing of flexible pavements, follow the so-called "general prescriptions" determined by specific standards. These standards favor the use of granular and stone materials for use in the asphalt coating layer. Such materials use granite and basalt crushed stone, having high cost and causing associated environmental impacts in all stages of production.

In this sense, the high demand for natural aggregates in pavement projects and the high cost associated with this product has caused researchers from all over the world to look for alternative materials that have physical, mechanical and environmental characteristics equal or superior to conventional aggregates.

In recent decades, interest in the application of industrial waste, steel slag aggregates in particular, in other areas has grown widely. This can be attributed to both economic and environmental issues. It should also be noted that the additional revenues, the reduction of costs involved in storage, in addition to the possibility of adding value through reuse, are reasons that enhance the proposal for reuse.

2. Materials and methods

The air-cooled ferronickel slag used in this research comes from a large mine located in the state of Pará. The (Fernandes, 2016).

In this context, there is a need to continue the search for new materials that in addition to mitigating the damage caused to the environment, offer a significant reduction in the cost of a paving project. Based on the above, the world's nickel reserves are estimated at 78,000,000 tons, with a production in 2016 of 2,250,000 tons. Brazil has the third largest nickel reserve in the world, with reserves estimated at 11,000,000 tons and production in 2019 of 67,000 tons. (USGS, 2020).

Ferronickel slag is a co-product generated from the production process of ferronickel alloys that are usually used in the manufacture of stainless steel and iron alloys. It can be classified into two different types, according to the cooling method, which can be aircooled slag or water-cooled (granulated) slag. Air cooled slag is slowly cooled outdoors in an open well, while water cooled slag is quickly cooled using water (Choi and Choi, 2015; Saha *et al.*, 2018).

Also, according to Saha and Sarker (2016), the chemical composition of ferronickel slag consists mainly of SiO₂, MgO and Fe₂O₃. This material is formed by amorphous silica, as well as crystalline minerals such as enstatite, forsterite and dropsied. The chemical compositions of the slag may be different depending on its source, processing, and cooling methods (Lemonis *et al.*, 2015; Maragkos *et al.*, 2009; Komnitsas *et al.*, 2007; Saha and Sarker, 2016).

It is important to highlight that the properties of ferronickel slag do not respect a general rule. Therefore, for each new deposit, its properties should be determined because they can vary depending on the origin and the method of processing the ore (Saha *et al.*, 2018).

It is important to highlight the differential of this research because it involves a new use of iron-nickel slag within the state of Pará, since almost all production is stored in industrial yards and therefore contributes to a significant environmental impact. In addition, data from the Highway Department indicate that predominantly natural aggregates are used to produce the asphalt mixture. This data highlights the importance of this study, which may lead to the reduction of stocks allocated in industrial yards.

material was sampled and transported to the Laboratory of Railways and Asphalt (LFA), in the Federal University of Ouro Preto (UFOP), where the tests were carried out. Figure 1 shows the ferronickel slag.



Figure 1 - Ferronickel slag received at Laboratory of Railways and Asphalt (LFA/UFOP).

The testing campaign of ferronickel slag was based on the physical, chemical, and environmental characterization of the slag.

The physical characterization of the slag was performed through the granulometry tests (DNER-ME 083/1998), relative density, bulk density and absorption of coarse aggregates (DNER-ME 081/1998), relative density of fine aggregate (DNER-ME 084/1995), relative density of filler (DNER-ME 085/1994), sand equivalent (DNER-ME 054/1997), form of aggregates (DNER-ME 086/1994), treton (DNER-ME 399/1999), durability (DNER-ME 089/1994), adhesion test (DNER-ME 078/1994) and abrasion Los Angeles (DNER-ME 035/1998).

In this study, the chemical characterization was performed using X-Ray Fluorescence to quantify the oxides present in ferronickel slag, while the X-Ray Diffraction and Scanning Electron Microscopy (SEM) with EDS analyzer tests sought the microstructural and morphological characteristics. These analyses were carried out with crushed and passed ferronickel slag samples in a sieve of either 200 or 75µm. In the case of chemical characterization tests there is no specific standard for conducting the tests. For this research, some references that proposed the use of steel slag for paving were the basis for the execution of the characterization. The main references were Fernandes (2016), Graffitti (2002), and Castelo Branco (2004). It is also necessary to differentiate the chemical characterization tests from those that intend to determine morphological and microstructural characteristics. In the environmental characterization, Leaching (NBR 10005/2004) and Solubility (NBR 10006/2004) tests were performed.

After the characterization, the specimens were molded and the stability and flow of the mixture was determined following the Marshall mix design according to DNER-ME 043/95 specification. Tensile strength by static diametral compression at 25 °C was done in accordance with the specification DNER-ME 138.

Through experimental procedures, the design of an asphalt mixture provides an optimal content of asphalt binder from a predefined particle size range.

The selection of the particle size range used was within the limits of the range C of DNIT for asphalt concrete, which is specified by DNIT-ES 031/2006.

The percentages of asphalt binder used in the mixtures were 4.0%, 4.5%, 5.0%, 5.5%, 6.0% and 6.5%. Three specimens were made for each content of asphalt mixture. The samples were used to determine the bulk specific gravity (Gmb), voids in the mineral aggregate (VMA), voids filled with asphalt (VFA), air voids (V_a) , stability and flow for each specimen.

For the calculation of optimum binder content, a graph is drawn with values in the mixture's binder content abscissa (%) and in the ordinates the values of air voids (V_a) and Voids Filled with Asphalt (VFA). Through the specification of the limits determined by DNIT for these evaluated parameters (V_a between 3% and 5% and VFA between 75% and 82%), four values of asphalt binder content are obtained through vertical lines originated from the specification. The design content is calculated as the average of the two central values of PAC (Bernucci *et al.* 2008).

3. Results and discussions

3.1 Physical characterization

All the laboratory tests of physical characterization and the project range

are presented below. Figure 2 shows the granulometry test of ferronickel slag.



Figure 2 - Granulometric curve of ferronickel slag.

Table 1 shows the final granulo-
metric distribution of the mixing proj-ect in accordance with DNIT Range
C with a composition of 12% coarseaggregate, 45% fine aggregate and
43% filler.

Sieve Size (mm)		% per sieve		Range C (DN	Project Range	
	Filler	Fine Aggregate	Coarse Aggregate	Lower Limit (%)	Upper Limit (%)	Passing (%)
37.5	0.0	0.0	0.0	100.0	100.0	100.0
25.4	0.0	0.0	0.0	100.0	100.0	100.0
19.1	0.0	0.0	0.0	100.0	100.0	100.0
12.7	97.9	2.1	0.0	80.0	100.0	89.8
9.5	33.2	66.6	0.1	70.0	90.0	84.6
4.8	0.9	96.9	2.2	44.0	72.0	58.7
2	0.0	36.9	63.1	22.0	50.0	33.3
0.42	0.0	20.5	79.5	8.0	26.0	13.1
0.2	0.0	21.4	78.6	4.0	16.0	7.8
0.075	0.0	18.0	82.0	2.0	10.0	4.6
Bottom	0.0	0.0	0.0	0.0	0.0	0.0

Table 1 - Project range of the ferronickel slag aggregate in the Range C of DNIT.

Table 2 shows the results of the other tests for characterization of ferronickel slag.

Tests	Results	Limits specified by DNIT ES 031 and IPR/1998	
Relative density – fine aggregates	3.340	-	
Relative density - coarse aggregates	3.199	-	
Relative density - filler	3.256	-	
Bulk density - coarse aggregates	2.975	-	
Water Absorption - coarse aggregates (%)	2.30	< 2	
Sand Equivalent – fine aggregates (%)	73.32	> 55	
Form of aggregates	0.72	> 0,5	
Treton (%)	21.9	< 60	
Durability (%)	0.77	< 5	
Adhesion test	Satisfactory	-	
Abrasion Los Angeles (%) (Range C)	47.9	< 50	

Table 2 - Results of characterization of ferronickel slag.

3.2 Chemical characterization

3.2.1 X-Ray fluorescence analysis

The result of the quantitative chemical analysis by X-ray fluores-

cence performed on the ferronickel slag sample is shown in Table 3.

Table 3 - Chemical species and their mass percentages.

Ferronickel slag	Chemical species (% by mass)								
	SiO ₂	MgO	Fe ₂ O ₃	Cr ₂ O ₃	SO ₃	CaO	MnO		
	47.77	30.75	17.31	2.00	1.14	0.47	0.41		

It is possible to observe that the chemical composition of the ferronickel slag studied is composed mainly of SiO_2 (silica), MgO (magnesium oxide) and Fe_2O_3 (iron oxide), which are the same components found

by Santos (2013), Wang (2016) and Saha and Sarker (2016).

3.2.2 X-Ray diffraction analysis

The results obtained for the X-ray diffraction test for ferronickel slag are

shown in Figure 3 and represent the occurrence of the mineralogical phases of the diffractometric standards.



Figure 3 - X-ray diffraction pattern of ferronickel slag.

The X-ray diffractogram obtained for the ferronickel slag sample shows a high incidence of peaks, indicating that the slag structure is predominantly crystalline. The diffractogram shows the presence of the mineral enstatite. Enstatite has a chemical formula (Mg,Fe)₂Si₂O₆, and is a mineral composed of silicon dioxide (SiO₃)

3.2.3 Scanning electron microscopy with EDS analyzer

Scanning electron microscopy (SEM) for the analysis and characterization of the different mineral phases in samples is used on a timely basis. Figure 4 shows the and magnesium oxide (MgO). Its hardness is approximately 5.5 and its bulk density is from 3.26 to 3.28. According to several authors, ferronickel slag basically consists of amorphous silica, as well as crystalline minerals such as enstatite (Lemonis *et al.*, 2015; Maragkos *et al.*, 2009; Komnitsas *et al.*, 2007).

morphological distribution obtained in

the analysis of the ferronickel slag sample

powder, so that it is possible to observe

the heterogeneity of the fines of the sample

The presence of the mineral enstatite in ferronickel slag is extremely beneficial from a mechanical perspective in asphalt mixtures, since, this mineralogical compound does not present expansive potential, such as steel slag. Therefore, its use in asphalt mixtures does not compromise the integrity of the pavement.

by the different contrast in the staining of the particles, an observation that can be confirmed by the analysis of its chemical composition performed in this same test.



Figure 4 - Indication of points found in typical morphology of pulverized ferronickel slag.

Figure 5 below shows the chemical composition of ferronickel slag considering the entire map, and not just pointedly.



Figure 5 - Indication of points found in typical morphology of pulverized ferronickel slag.

Regarding the result presented in Figure 5, the predominance of the pink color is highlighted, which represents the predominance of silica in

the sample. This data corroborates the results already presented of x-ray dif-

fraction that pointed out mineralogical compounds related to silica (enstatite).

Table 4 shows the result of the quantitative analysis by microscopy

with EDS analyzer considering the entire sample.

Ferronickel slag	Chemical species (% in oxide content)							
	Si	Mg	Fe	Al	Cr	Ca	Mn	
Sum of maps	51.3	29.3	13.4	3.5	1.7	0.4	0.4	

Table 4 - Results of the quantitative analysis performed.

The results of the quantitative analysis by scanning electron microscopy performed with EDS analyzer on ferronickel slag were very close to the results found in the quantitative analysis performed by X-ray fluorescence, reaffirming the compounds previously found.

3.3 Environmental characterization

The Leaching test shows that there are no leached elements above the limits established by NBR 10004/2004 for ferronickel slag.

The Solubilization test did not show the presence of leached elements above the limits established by NBR 10004/2004.

According to the specifications of the

3.4 Marshall mix design

To determine the design content of the research, all volumetric parameters of mixtures with asphalt content of 4.0%, 4.5%, 5.0%, 5.5%, 6.0% and norms NBR 10004/2004, 10005/2004 and 10006/2004, it can be pointed out that the ferronickel slag is classified as Class IIB (Non Hazardous - Inert) material because it does not have any parameter in the sample solubilized in concentrations higher than the standards of water potability. And it can also be considered that the sample is not corrosive and not reactive because it does not have the characteristics that indicate such properties.

This result is extremely positive for the use of ferronickel slag in asphalt mixtures. Since the material does not leach compounds and does not solubilize, it will not present impacts to the environment.

6.5 % were calculated.

With the determination of the volumetric parameters V_a and VFA for each specimen, it was possible to construct a graph with the abscissa represented by the percentage of asphalt binder by weight and the ordinates being the values of V_a and VFA, as shown in Figure 6.



Figure 6 - Curve for determining the optimum asphalt binder content.

Based on this graph, the optimum asphalt binder content was defined, and the chosen value should be among the central values of asphalt binder content. Therefore, the optimum asphalt binder content defined for the mixture was 6.00%, which is the lowest whole number within the range indicated by the Va and VFA limits (5.95% to 6.35%).

After determining the volumetric mea-

surements and choosing the design content, the mechanical stability and flow parameters for optimum asphalt binder content of 6.0% were obtained through the Marshall press, which are presented in Table 5.

Binder Content (%)	Stability (N)	Flow (mm)
6.0	20558.67	5.50
6.0	20856.17	6.00
6.0	22282.98	5.50
Mean (N)	21232.61	5.67
Standard deviation(N)	752.59	0.24

Table 5 - Results of stability and flow tests for the optimum asphalt binder content of 6.0%.

Table 6 shows the limits imposed by DNIT 031/2006, as well as the results found in this research.

Table 6 - Result of dosing requirements of hot mix asphalt by DNIT 031/2006.

Optimum asphalt binder content of 6.0%							
	Procedure	Results	Limits				
V _a (%)	DNER-ME 043	4.76	3 a 5				
VFA (%)	DNER-ME 043	77.44	75 - 82				
Stability (minimum) (N) (75 golpes)	DNER-ME 043	21232.61	4903.32				
Tensile strength by static diametral compression at 25 ° C, minimum (MPa)	DNER-ME 138	1.62	0.65				

From the results of the volumetric parameters obtained for the mixture of hot mix asphalt with the addition of ferronickel slag aggregate, it is possible to state that the mixture can be executed

because it fits all the parameters recommended by DNIT 031/2006.

As a proposal for future study, the researchers intend to analyze the same ferronickel slag using dynamic tests such as the resilient modulus and fatigue. This change aims to study the elastic behavior of the material in the face of traffic demands, instead of the plastic behavior.

4. Conclusions

The study presents the characterization of the ferronickel slag produced in the state of Pará for reuse in asphalt mixtures and the corresponding analysis of its performance against the limits set by Brazilian standards. One of the main differentials of this research is the reutilization of ferronickel slag in asphalt mixtures in a Brazilian state with a large part of its unpaved road network and a lack of natural inputs for the execution of the works. Therefore, an attempt is being made to solve a problem in the expansion of the state highway network with the use of waste that had the industrial vards as its main destination, causing environmental problems.

Based on the analysis of the results presented, it can be stated that the physical characterization of the aggregates obtained conformity in almost all the tests performed. The exception was the water absorption test which resulted in 2.3% when the limit is 2%. This result is explained by the high porosity of the slag, however, does not compromise its mechanical competence as evidenced in the tests.

Concerning the chemical and morphological characterization, it can be reported that there is a clear predominance of SiO₂ (silica), MgO (magnesium oxide) and Fe₂O₃ (iron oxide). In addition, the presence of the mineral enstatite was observed in the morphological analysis. These compounds do not present an expansive potential for ferronickel slag, so their use in asphalt mixtures will not affect the integrity of the pavement under saturated conditions.

Through the environmental characterization, it was possible to perceive that the aggregate does not represent risks to the environment, as it is a class IIB (Non Hazardous / Inert) waste. From the results of the volumetric parameters obtained for the mixture of hot machined bituminous concrete with the addition of ferronickel slag aggregate, an optimum asphalt binder content of 6.0% was obtained.

In an economic and technical analysis, the higher consumption of asphalt binder by ferronickel slag compared to other natural aggregates should be mitigated by its final cost being much lower than the natural aggregate. Therefore, in order to pave roads in the state of Pará, ferronickel slag has a lower cost than conventional aggregate.

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