Investigating mechanical properties of hot mix asphalt containing iron ore flotation tailing
Análise do comportamento mecânico de uma mistura asfáltica contendo rejeito de flotação de minério de ferro
Análisis del comportamiento mecánico de una mezcla asfáltica conteniendo relaves de flotación de mineral de hierro

Abstract
Studies involving the use of iron ore tailings from the beneficiation process in different market niches are increasingly necessary due to the large environmental damage caused by their disposal in dams, since they are waste generated in high volumes and which have low economic value for mining companies. The main purpose of this study was to investigate mechanical properties of hot mix asphalt mixture containing iron ore flotation tailing. For this, a mixture containing iron ore flotation tailing and a control mixture with natural aggregates and stone dust were prepared according to the Marshall methodology and tests of stability and flow, indirect tensile strength, resilient modulus, fatigue test and dynamic creep tests were conducted. Results show that the mixture with iron ore flotation tailing had performed technically appropriate, due to the similarity of mechanical properties of the control mixture. Therefore, the results obtained revealed that the iron ore flotation tailing presented technical characteristics appropriate to its use incorporated into hot mix asphalt. In addition, their use as an alternative material in asphalt pavements can generate environmental benefits by reducing the volume of material deposition in tailings landfills and exploration areas for mineral aggregate mining.

Keywords: Pavements; Asphalt mixtures; Iron ore flotation tailings.

Resumo
Estudos envolvendo a utilização de rejeito de flotação resultante do processo de beneficiamento do minério de ferro, em diferentes nichos de mercado, se mostram cada vez mais necessários diante do grande passivo ambiental causado pela sua disposição em ateros, uma vez que se tratam de resíduos gerados em grandes volumes e que apresentam baixo valor econômico para as empresas mineradoras. O objetivo desse trabalho foi analisar o comportamento
Iron ore tailings (IOT) are a form of solid waste produced during the beneficiation process of iron ore concentrate. Among all kinds of mining solid waste, IOTs are one of the most common solid wastes in the world due to their high output and low utilization ratio (Tang, et al., 2019; Sun, et al., 2011). For this reason, the production of tailings has been getting much attention of companies around the world, to minimize environmental impacts and costs associated with the processes of deposition, with the use of mine tailings in civil engineering construction (Kuranchie, et. al., 2014). This practice can help reduce the emission of greenhouse gases, by avoiding the mining of virgin engineering materials, providing cheaper alternative materials for building and construction, and for natural resource conservation (Chen, et al., 2011).

The IOT are generated by the ore beneficiation process and the well-now process are flotation separation and magnetic separation. The flotation process consists of a surface physical-chemical process, used in the separation of minerals, which gives rise to the formation of an aggregate, mineral particle and air bubble, which, in a watery environment, floats in the form of foam, while the magnetic concentration process consists of the tailings from the processing of waste introducing dry treatment, without using water to process it (Filippov, et al., 2014).

Roads are built using different materials like gravel, sand, aggregates, asphalt binder, cement, etc. There has been
constant research to replace or substitute the materials by other materials for better pavement of roads. In the process, the waste produced from various sources can be an effective replacement, as the waste will be available at free of cost or with minimum price and on the other hand, handling and disposal of waste also minimizes (Gayana & Chandar, 2018). As a measure for conserving natural resources and environmental pollution, the use of waste materials in highway construction has been considered in recent years, and different types of waste materials have proved applicable as a replacement for virgin materials (Taherkhani & Arshadi, 2019).

Applying iron tailings to road engineering can consume a lot of iron tailings and reduce the cost of road engineering. China's iron tailings as road building materials are still in initial study phase (Bing et al., 2018). The use of waste materials in asphalt pavements is the most effective way for decreasing the exploitation of natural resources and environmental pollution (Arabani et al., 2017).

A few studies have utilized IOT with material in asphalt pavements. Bastos et al. (2016) evaluate the viability of iron ore tailings from tailing dams as an alternative material for road infrastructure. The iron ore tailings were characterized according to their chemical, mineralogical, environmental, and physical properties. Subsequently, the tailings were chemically stabilized using cement, lime, or steelmaking slag as binder. The tailing-cement, tailing-lime, and tailing-slag mixtures studied consisted of 1, 2, 5, and 10% binder content, each. The characterization methodology included chemical and mineralogical analysis through X-ray fluorescence and diffraction; environmental analysis with leaching and dissolution tests; evaluation of compaction curves; California bearing ratio (CBR) tests and expansion assessment; compressive strength, with curing in moisture chamber or at open air; water absorption; and evaluation of durability of the mixtures. The cement was the most efficient stabilizer among the studied binders.

Lara et al. (2018) studied the mechanical behavior of a lateritic soil with addition of iron ore flotation tailings for application as subgrade reinforcement material and subbase of road pavements, considering the substitution of 0%, 25%, 50% and 75% of the IOT on in the mixtures with the soil. The results showed an increase in soil CBR values with addition of IOT, and a decrease in the expansion. Results of cyclic triaxial tests confirmed the capability of the resilient composite model applied to the mixtures, which enable them to be used as pavement layers, especially as subgrade reinforcement material and subbase material. Grasse et al. (2019) studied the effect of the addition of the iron ore flotation tailings on a lateritic soil chemically stabilized with 2%, 4% and 6% of lime, for application in base and subbase layers of road pavements. The results showed an increase in CBR values of the soil-lime mixtures with the addition of iron ore flotation tailing, and the decrease of the expansion. It was concluded that the IOT addition caused positive changes in the values of unconfined compression strength of the soil-lime mixtures.

Apaza et al. (2018) studied the physical and mechanical characterization of cold-mix microsurfacing treatment, considering the substitution of 0%, 10%, 15% and 20% of the iron waste as small aggregate. It was conducted the Wet Stripping Test – WST and the Modified Cohesion Test – MCT to analyze the compatibility of materials. The mechanical behavior of cold-mix microsurfacing test specimens containing aggregates of the iron residue showed to perform similarly to the usual aggregates, until 20% of substitution, indicating that it is a material with promising use in this type of technique of paving.

A review of the literature on IOT as material in asphalt pavements shows that the properties of these mixtures, especially hot mix asphalt mixtures, have not been fully investigated. Therefore, in this laboratory investigation, the impacts of utilizing IOT, in some engineering properties of hot mix asphalt (HMA) were investigated.

2. Materials

The materials utilized in this research were asphalt binder, aggregates, and iron ore flotation tailing (IOT). A asphalt
binder of 50/70 penetration grade from Gabriel Passos Refinery (REGAP) in Brazil was used to produce the specimens in this study. Table 1 shows a summary of the asphalt binder properties.

Table 1. Conventional rheological properties of the asphalt binder used in this study.

| Test                          | Standard  | Results | Specification limits |
|-------------------------------|-----------|---------|----------------------|
| Penetration Index (25°; 0.1 mm) | ASTM D5   | 50      | 50 to 70             |
| Softening point (°C)          | ASTM D36  | 50      | ≥ 46                 |
| Flash point (°C)              | ASTM D92  | 362     | 235                  |
| Viscosity at 135°C (Pas)      | ASTM D4402| 0.302   | ≥ 0.274              |
| Ductility (25°; cm)           | ASTM D113 | >150    | ≥ 60                 |
| Specific gravity (25°; g/cm³) | ASTM D70  | 1.011   | N/A                  |

Source: Authors (2021).

In this study, gneiss aggregates from Itabira region, Minas Gerais State, were collected and used in asphalt mixtures. Table 2 presents the properties of the coarse, fine, and filler fractions of aggregates and filler fractions of the IOT. IOT samples were collected at the output of the flotation duct of the Cauê mine from Itabira, Brazil.

Table 2. Aggregates and filler IOT properties.

| Properties/ Materials | Bulk specific gravity (g/cm³) | Los Angeles Abrasion Test (%) | Sand equivalent (%) | Flat and elongated particles (%) | Plasticity index ASTM-D4318 | Loss in sodium sulphate solution (%) | Source: Authors (2021). |
|------------------------|-------------------------------|------------------------------|---------------------|---------------------------------|-----------------------------|-------------------------------------|
| Coarse aggregates      | 2.660                         | 42                           | 24                  | -                               | -                           | 1.42                                |
| Fine aggregates        | 2.717                         | -                            | 74                  | -                               | NP                          | 3.22                                |
| Filler                | 2.759                         | -                            | -                   | NP                              | -                           | -                                   |
| Filler (IOT)           | 3.160                         | -                            | -                   | NP                              | -                           | -                                   |

A dense gradation with the maximum aggregate size of 19.1 mm was selected based on the Brazil Asphalt Pavement Standard for asphalt mixtures, denominated C granulometric composition. Figure 1 illustrates the specification limits and gradation of the mixtures in this study. Figure 2 shows the granulometric curve of the IOT, utilized in this study.
As shown in Figure 1, the particle size range chosen for the mixtures is in the center of the limits of the range C granulometric composition.

According to Figure 2, the iron ore flotation tailing has more than 90% passing material in the 0.18 mm sieve and approximately 50% passing material in the 0.075 mm sieve.

Figure 3 shows the IOT used in the experiment. The mineralogical composition of the IOT was determined by X-Ray Diffraction and the results indicate that the iron ore flotation has mineralogy composed of silicates (quartz, kaolinite, talc, pyrophyllite and muscovite), iron oxide (hematite) and aluminum hydroxide (gibbsite). Additionally, particle shape and surface analysis of the IOT fillers were examined by scanning electron microscopy (SEM). As shown in Figure 4, particles had angular shapes and there is a heterogeneity in the shape and size of the particles, ranging from particle sizes between 10 to 100 µm.
3. Method

3.1 Specimens preparation

The asphalt mixtures were prepared according to ASTM D1559. The aggregates were heated to 150–160°C for 24 h before preparing HMA mixtures. Then, the asphalt binder was heated to the temperature of 145–151°C before being mixed with aggregates. The amount of filler for all studied mixture proportions was fixed to 6%. The optimum asphalt binder contents were found to be 4.72% to the control mixture (CM) and 5.26% to the mixture with IOT (IOTM), using the Marshall mix design method. The mixtures were compacted at 135-140°C by a Marshall hammer with 75 blows per side and three samples were prepared to each test.
3.2 **Marshall Tests**

Marshall tests were conducted on the specimens according to ASTM D1559 standard method. The prepared specimens were placed in a water bath at 60°C for 30 min of immersion, after which they were loaded using a Marshall test set-up at the constant rate of 50.8 mm/min. The force required for breaking the specimen was measured as the Marshall stability (MS), and the diametrical deformation of the specimen at failure was measured as Marshall Flow (MF).

3.3 **Indirect Tensile Strength (ITS)**

Indirect Tensile Strength (ITS) tests were conducted according to ASTM D6931 standard method (specimens with 101.6 mm in diameter and 63.5 mm in height). Three specimens for each mixture were prepared and placed in a conditioned chamber at 25°C per 4 hours, after which they were loaded using a Marshall test set-up at the constant rate of 50.8 mm/min. The required force for breaking the specimen was measured and ITS was calculated using the Equation (1),

\[
\text{ITS} = \frac{600P}{\pi D t}
\]  

where ITS is the indirect tensile strength in kPa, P is the maximum applied load for breaking the specimen in N, D is the specimen diameter in mm, and t is the specimen height immediately before test in mm.

Indirect tensile strength is considered as the potential test method for determining the tensile properties of asphalt mixture which can be further related to rutting and cracking properties of asphalt mixture (Moghaddam, et al., 2014). The values of ITS may be used to evaluate the relative quality of asphalt mixtures in conjunction with another laboratory mix design testing and for estimating the potential for rutting or cracking. Tensile strength is one of the main properties of asphaltic mixtures and is related to the strength against cracking and permanent deformation. A mixture with a higher tensile strength is more resistant against cracking and permanent deformation (Esfahani & Jahromi, 2020; Taherkhani & Arshadi, 2019).

3.4 **Resilient Modulus (Mr)**

The Mr test was conducted using a modular electro-mechanically operated asphalt tester apparatus according to ASTM D4123. A haversine load pulse was applied at a frequency of 1 Hz, including 0.1 s loading and 0.9 s rest period. The Poisson’s ratio was assumed as 0.35. Based on the standard method, depending upon the loading frequency, mix type, and testing condition, 50–200 loading repetitions might be needed. The minimum value must be determined so that the resilient deformations become stable. Based on the laboratory observations, 150 load repetitions were selected. In this research, to measure the resilient modulus the specimens were tested at three temperatures of 10, 25 and 40°C. Three specimens were fabricated for each test. For an applied dynamic load of P in which the resulting horizontal dynamic deformations are measured, the total Mr value can be calculated using Equation (2),

\[
M_r = \frac{P(t+0.17)}{\Delta H}\n\]  

where Mr is the resilient modulus (MPa), P is the repeated load (N), t is the sample’s thickness (mm), ΔH is the recoverable horizontal deformation (mm) and ν is the resilient Poisson ratio.

The values of resilient modulus can be used to evaluate the relative quality of materials as well as to generate input for pavement design or pavement evaluation and analysis. Resilient modulus, which describes the elastic and plastic behavior of a mixture against loading, shows a pavement’s response in terms of dynamic stresses and the corresponding strains. Since this parameter is a good indicator of real mixture performance, many have used it in pavement design in recent decades (Esfahani & Jahromi, 2020).
3.5 Creep Dynamic Test

In this study, dynamic creep tests were conducted on cylindrical specimens (101.6 mm in diameter and 63.5 mm in height). During the test dynamic compressive loadings with the peak of 200 kPa were applied. The loading time was 0.1 s and rest period of 0.9 s was designated for this study. The amounts of cumulative permanent strains were recorded during applying 3600 loading cycles. The cumulative axial strain was calculated according to Equation (3),

\[ \varepsilon = \frac{h}{H_0} \]  

(3)

where \( \varepsilon \) is the cumulative axial strain, \( h \) is the axial deformation and \( H_0 \) is the initial height of specimen. Dynamic creep test was conducted at 40 °C, and to reach a uniform mixture temperature all the specimens were placed in controlled temperature chamber for at least 4 h.

Creep modulus is the most important output of the dynamic creep test. The value of creep modulus is an additional indication of the resistance to permanent axial deformation and for bituminous specimens which are basically obtained from the ratio of applied stress (200 kPa) to the cumulative compressive strain at a defined temperature and time of loading. Mixtures with a lower creep modulus are known to undergo higher deformation (Nejad, et al., 2014). The creep modulus (S) was calculated according to Equation (4),

\[ S = \frac{\sigma}{\varepsilon} \]  

(4)

where \( \sigma \) is the applied stress and \( \varepsilon \) is the axial strain.

3.6 Fatigue Test

One of the important parameters to be considered in pavement design is the traffic and the repeated load applications by the moving vehicles. Excessive deflection of pavement under moving load is considered as one of the prime mechanisms of pavement failure. The dynamic stiffness of the pavement mix plays an important role in determining the fracture characteristics and fatigue behavior of the pavement (Chandra, et al., 2002). Fatigue tests are carried out with two models: controlled strain and controlled stress. In the controlled strain mode, the strain is kept constant by decreasing the stress during the test, whereas in the controlled stress mode, the stress is kept constant, which increases the strain during the test (Arabani, et al., 2010).

In this study the tests were conducted under controlled stress conditions. Fatigue tests were conducted at 10, 20, 30 and 40 % of ultimate compressive indirect tensile strength. Three specimens (101.6 mm in diameter and 63.5 mm in height) for each mixture and each level of indirect tensile strength were prepared and placed in a conditioned chamber at 25°C for 4 hours (Figure 5). The loading time was 0.1 s, and 0.9 s was considered for rest period. Failure of mixes occurred when the specimen collapsed.
Figure 5. Electro-mechanically asphalt tester apparatus for fatigue tests.

Source: Authors (2021).

A Linear regression analysis of the fatigue tests results was used to determine fatigue functions for the asphalt mixtures using the Equation (4),

\[ N_f = a \left( \varepsilon_0 \right)^b \]  

where \( N_f \) is the number of cycles to failure of the specimen, \( \varepsilon_0 \) is the initial tensile strain (micro-strain), and \( a \) and \( b \) are experimentally determined coefficients.

4. Results and Discussion

4.1 Marshall Stability and Flow Tests

Table 3 presents the Marshall Stability and flow results for the CM and IOTM mixtures. The values represent the average of the results for three replicates specimens of each mixture. The results show that the mixtures containing IOT have a higher stability than the CM mixture. As shown in the Table 3, Marshall stability of the IOTM specimens increased 5.3% and the flow decreased 6.8%. High flow values generally indicate a plastic mix that will experience acceleration permanent deformation under traffic loads.

Table 3. Marshall Stability, Flow and MQ for all mixtures.

| Mixture ID | Marshall stability (kN) | Marshall flow (mm) | MQ (kN/mm) |
|------------|-------------------------|--------------------|------------|
| CM         | 15.15                   | 2.94               | 5.15       |
| IOTM       | 15.96                   | 2.74               | 5.82       |

Source: Authors (2021).

The relationship between MQ (Marshall Stability divided by Flow) and type of mixture is provided in Table 3. As shown in the Table 3, MQ of IOTM specimens increased 13.03%. According to Arabani, et al. (2010) and Nejad, et al. (2014) Marshall quotient (MQ) is defined as the ratio of stability (kN) to flow (mm) and a higher value of stability divided by flow indicated a stiffer mixture and, hence, the mixture was likely to be more resistant to permanent deformation.

4.2 Indirect Tensile Strength (IDT)

Table 4 presents IDT results for the CM and IOTM mixtures. The values represent the average of results for three replicates specimens. The results show that the mixtures containing IOT have a similar value that the obtained for CM mixture.
According to the Brazil National Department of Transport Infrastructure Highway Standard Code (DNIT 031-2006), minimum IDT must be 0.65 MPa in hot mix asphalt.

| Table 4. Indirect Tensile Strength results. |
|------------------------------------------|
| **Mixture ID** | **IDT (MPa)** |
| CM | 1.431 |
| IOTM | 1.434 |

Source: Authors (2021).

### 4.3 Resilient Modulus (Mr)

Figure 6 illustrates the resilient modulus variation versus temperature for CM and IOTM specimens. Each specimen was prepared with optimum binder content and the results presented in Figure 6 are the average of three replicates, for each temperature. The results show that as the temperature increases, the resilient modulus of the asphalt mixtures decreases. This phenomenon occurs due the change in the viscosity of bitumen with respect to temperature (Meor & Teoh, 2008).

For the three temperatures tested, the IOTM asphalt mixture presented lower values of resilient modulus compared with those results for the CM mixtures. However, for the temperature of 10 °C the resilient modulus of the IOTM was lower by about 14.5% in comparison to the control mixture. For temperatures of 25 °C and 40 °C the reductions were 2.82% and 6.95%, respectively.

![Figure 6. Resilient modulus versus temperature.](image)

Source: Authors (2021).

### 4.4 Creep Dynamic Test

Creep modulus and cumulative axial strain values of control and IOT mixtures are given in Figure 7. The values are the average of three tests results by each mixture. In the CM specimens, the creep modulus was lower than the results for the IOTM, while the cumulative axial strain was higher.

To the same loading level and test conditions, the IOT mixture showed less susceptibility to permanent deformation, and compared with the MQ results, the IOTM showed as a stiffer mixture and more resistant to permanent deformation. According to the correlations between traffic intensity and creep modulus, presented by Little, et al. (1993), the values found
for the mixtures are compatible with the performance of asphalt mixtures for application in pavements subject to moderate traffic intensity.

**Figure 7.** Creep modulus and cumulative axial strain values of the control and modified asphalt mixture with IOT.

![Figure 7](image)

Source: Authors (2021).

### 4.5 Fatigue test

As shown in Figure 8, the fatigue prediction models for mixtures were presented according to the fatigue tests results obtained in this study. There is a linear relationship between tensile strain and fatigue life in the logarithm scale. The fatigue models for mixtures and R2 values are presented in Table 5. Although the models show similar behavior, for the initial application stages of load cycles the mixture with the addition of IOT showed higher values of strains, and for the final stages the same mixture showed lower values, when compared with the CM.

**Figure 8.** Comparison of the curves for the fatigue life of CM and IOT mixtures.

![Figure 8](image)

Source: Authors (2021).
Table 5. Fatigue prediction models.

| Mixture ID | Fatigue Model | R^2  |
|------------|---------------|------|
| CM         | \( N = 2 \times 10^{-10} \times \left( \frac{1}{T} \right)^{2.306} \) | 0.964 |
| IOTM       | \( N = 7 \times 10^{-9} \times \left( \frac{1}{T} \right)^{2.908} \) | 0.967 |

Source: Authors (2021).

R^2 value determine how closely the data conform to a linear relationship. The higher the R^2, the better the model fits the data. As presented in Table 5 the R^2 values to the mixtures fit to the data and indicates that the models explain all the variability of the response data around its mean.

5. Conclusions

In this research, the impacts of utilizing IOT in some engineering properties of hot mix asphalt (HMA) were investigated. Based on the results, the following conclusions can be derived:

- When IOT was used in the mixture, Marshall Stability average value of the specimens increased, and flow decreased.
- IOT mixture had a higher value of stability divided by flow (MQ), which indicated a stiffer mixture, and the mixture was likely to be more resistant to permanent deformation.
- Although the MQ higher value obtained for the IOT mixture, the indirect tensile strength tests result in similar average values for both.
- At 10, 25 and 40°C temperatures the highest resilient modulus values of the mixtures were obtained from the CM mixture.
- At 10°C, the resilient modulus of bituminous mixtures with IOT exhibited lower value. This shows that a better performance can be achieved in cold region by using IOT as filler.
- According with the results of creep tests, resistance against permanent deformation of IOT asphaltic mixtures increases.
- for the initial application stages of load cycles, the mixture with IOT showed higher values of strains, and for the final stages the same mixture showed lower values, when compared with the CM.

Based on the results, as a way of conserving natural resources and reduce high accumulation of this material in the environment, the studied iron ore tailing material can be used in HMA for road construction.

As a suggestion for future work, build experimental segments in roadways with the studied mixtures to evaluate their performance under the action of vehicular traffic loads and the weather, over design time.

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