Contribution to the non-destructive evaluation of bituminous mixes 
by electrical measurements

Article Info:
Article history: Received 2023-07-28 / Accepted 2023-10-04 / Available online 2023-10-04 
doi: 10.18540/jcecvl9iss8pp16003-01eup

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Abstract
The incorporation of additives into bituminous mixes is a very important technique for improving the mechanical and electrical properties of the pavement body. The purpose of this work is to present and interpret the different results of the tests carried out in the laboratory; such as FENIX direct tensile test and electrical resistivity test. In this work, we will present comparative studies between the mechanical and electrical characteristics of bituminous mixes based on conductive additives (steel fibers and graphite powder). This comparison makes it possible to classify these resulting materials as materials that have the ability to respond to different multifunctional applications.

Keywords: Asphalt concrete. FENIX test. Electrical resistivity. Conductive additives. Mechanical and electrical properties.

1. Introduction
A variety of multipurpose applications, including heated pavements for the removal of snow and ice, self-sensing, self-healing, and energy recovery, may be satisfied by conductive asphalt (Rew et al., 2017; Park et al., 2014; Wang et al., 2016). The conductive bituminous mix can be defined as a mixture of bitumen, aggregates and electrically conductive additives (García et al., 2009; Wu et al., 2010), to obtain the best electrical properties without affecting its mechanical performance (Liu et al., 2021). In order to transmit electrical conductivity and modify the characteristics of bituminous mixes, researchers have tested various conductive fibers and powders, including steel fibers (Wang et al., 2016; Solanki et al., 2011; Lau et al., 2020; Paluri et al., 2020), carbon fibers (Wu et al., 2013; Slebi-Acevedo et al., 2019), wool steel (García et al., 2009) and graphite powder (Rew et al., 2017; Park et al., 2014). Previous research shows that fiber reinforced...
asphalt mixes develop good fatigue cracking resistance and flexural cracking resistance (Wang et al., 2016). Adding fibers to bituminous mixes ensures their stability and their mechanical strength (Lau et al., 2020; Paluri et al., 2020; Slebi-Acevedo et al., 2019; Xiong et al., 2015) and increases their dynamic modulus and even their fatigue and ductility behavior (Slebi-Acevedo et al., 2019). An adequate amount of fibers modifies the properties of the bituminous mix, thus reducing penetration and increasing the softening point. The added fiber also modifies the viscoelasticity of the bitumen (Slebi-Acevedo et al., 2019; Mohammed et al., 2018). In addition, the fibers have been considered as a positive reinforcement material for bituminous mix (Slebi-Acevedo et al., 2019).

In asphalt mix, fibers have been added to prevent drainage in porous asphalt mix and stone matrix asphalt mix (Park et al., 2015), to improve rutting resistance (Solanki et al., 2011). The conductivity of bituminous mixes is proportional to the content of added fibers (Wang et al., 2016) and conductive powders (Hasan et al., 2021). The capacity for self-sensing of tension and self-control of compressive stresses is greater in bituminous mix that contains graphite (Wang et al., 2017). The conductive property can be greatly improved by adding carbon additives in the form of graphite powder (Liu et al., 2009). The conductive bituminous mix with graphite has a higher fatigue resistance than the ordinary bituminous mix (Wu et al., 2010). Graphite increases thermal conductivity (Wang et al., 2017) and also increases electrical conductivity (Park et al., 2014). The addition of graphite as an electrically conductive additive significantly improves the resistance to rutting, increases the dynamic modulus and considerably reduces the resistance to indirect tensile cracking of the bituminous mix (Huang et al., 2009).

The goal of this study is to assess how steel-fibers and graphite powder affect the mechanical properties and electrical conductivity of asphalt concrete. Additionally, it establishes a connection between electrical and mechanical properties. The performance of the conductive bituminous concrete is evaluated using two tests: the direct tensile test (FÉNIX test) and the electrical resistivity test.

2. Experimental work
2.1 Materials

According to the Marshall design method, half-grained asphalt concrete of the 0/14 class was achieved. The gradation curve is depicted in Figure 1. It is possible to determine percentages of the mixture that must be inserted in reference spindle 0/14 based on different aggregate grain sizes. The percentages obtained are shown in Table 1.

![Figure 1 – Gradation Curve.](image-url)
Table 1 – Mixture Percentages.

<table>
<thead>
<tr>
<th>Binder and Aggregates</th>
<th>Percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8/15</td>
<td>37</td>
</tr>
<tr>
<td>3/8</td>
<td>22</td>
</tr>
<tr>
<td>0/3</td>
<td>37</td>
</tr>
<tr>
<td>Fillers</td>
<td>04</td>
</tr>
<tr>
<td>Bitumen</td>
<td>6</td>
</tr>
</tbody>
</table>

The asphalt and aggregate properties are shown in Table 2. The qualities of bitumen include: penetration at 25°C (P): 43.5 (1/10) mm; softening point temperature (T-R-B): 52 °C.

Table 2 – Properties of aggregates.

<table>
<thead>
<tr>
<th>Aggregates</th>
<th>(0/3)</th>
<th>(3/8)</th>
<th>(8/15)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cm³)</td>
<td>2.72</td>
<td>2.72</td>
<td>2.72</td>
</tr>
<tr>
<td>Methylene blue (ml/g)</td>
<td>0.76</td>
<td>/</td>
<td>/</td>
</tr>
<tr>
<td>Micro drop (%)</td>
<td>/</td>
<td>/</td>
<td>13.8</td>
</tr>
<tr>
<td>Los - Angeles (%)</td>
<td>/</td>
<td>/</td>
<td>19.11</td>
</tr>
<tr>
<td>Friability of sand (%)</td>
<td>39.92</td>
<td>/</td>
<td>/</td>
</tr>
<tr>
<td>Sand equivalent (%)</td>
<td>79.34</td>
<td>/</td>
<td>/</td>
</tr>
<tr>
<td>Electrical resistivity (µcm)</td>
<td>&gt; 10^{14}</td>
<td>&gt; 10^{14}</td>
<td>&gt; 10^{14}</td>
</tr>
</tbody>
</table>

Conductive additives: The steel fibers used in this study vary in size between (0.8 and 1.2 cm) in length and 0.2 in diameter (see Figure 2), with a tensile strength of 501 MPa, an electrical resistivity of 8. 10^{-7} (Ω. m) and with a Young’s modulus of 1346 MPa. And the graphite powder is less than or equal to 80 μm manually crushed (Figure 3).

![Figure 2 – Steel fibers.](image)

![Figure 3 – Graphite powder.](image)

Sample preparation: cylindrical specimens 100 mm in diameter and 60 mm long (Figure 4) were used for the tests. The binder, graphite powder, aggregates, and steel fibers were heated at 150°C for 120 minutes. They were combined into the heated vessel of a mixer and mixed until the graphite powder, steel fibers, and aggregates were completely coated and evenly distributed with the binder.

In compaction molds, the mixture was compressed using 50 strokes on each side. Before testing, samples taken out of the molds were kept in the lab environment for 24 hours. FENIX direct tensile test specimens containing 1.0%, 3.0%, 5.0%, 10.0%, 15.0%, 20.0%, 25.0%, and 30.0% graphite powder by volume of bitumen and 0.20 %, 0.40 %, 0.60 %, 0.80 %, 1.00 %, and 1.20 % steel fibers by weight of the blend.
2.2 Test methods

FENIX test (direct tensile test): The test was carried out by a FENIX device (Figure 5). The Marshall test specimens were divided into two with a gap of 6 mm and fixed in FINEX plates, it is necessary to apply a displacement at a constant speed (01 mm/min). The data acquisition system recorded the tensile strength and the vertical displacement.

Equation (1) is used to calculate the samples’ post-test tensile strength (Bitencourt et al., 2019):

\[ R = \frac{2F}{\pi \theta h} \]  

(1)

Where R is Tensile strength (Pa), \( \theta \): Diameter of the specimen (m), F: Total vertical load applied (N), and h: Height of the specimen (m).

The dissipated energy \( G_D \) is calculated by equation (2):

\[ G_D = \frac{W_D}{h.\theta} \]  

(2)

Where \( G_D \) is Energy dissipated (J/m\(^2\)) and \( W_D \): Work dissipated during the test application (kN mm).

\[ W_D = \int_{0}^{\Delta R} F \cdot du \]  

(3)
The ductility modulus is the displacement at 50% of the maximum post-peak force, which is an indication of the ductility of the bituminous mixture. It designates the ability of a material to deform plastically without breaking.

The ductility modulus (mm) is calculated as follows:

\[ M_d = \Delta_d \left( \frac{F_{\text{max}}}{2} \right) \]  \hspace{1cm} (4)

Where \( \Delta_d \) is Movement at 50% of maximum strength and \( F_{\text{max}} \) : Maximum strength.

2. Electrical resistivity test

In this study, electrical resistance was measured below \( 40.10^6 \, \Omega \) using a CHAUVIN ARNOUX megohmmeter and above this value using a multimeter. At the two ends of the cylindrical specimens, two electrodes made of copper plates were connected and positioned.

![Figure 6](image)

Figure 6 – Measures electrical resistance using the two-point method.

Ohm's second law allowed for the calculation of electrical resistivity after measuring the resistance (Bouazza et al., 2018)

\[ \rho_E (\Omega \cdot \text{m}) = R (\Omega) \times S (\text{m}^2)/h (\text{m}) \]  \hspace{1cm} (5)

Where \( \rho_E \) is Electrical resistance, \( h \): Height of the specimen, \( R \): Measured resistance, and \( S \): Conductive surface of the electrode.

3. Results and discussion

3.1 Comparison between the mechanical properties of bituminous mixes

Figure 7 illustrates the variation in the energy dissipated as a function of the ductility modulus of the two additives (steel fiber and graphite powder). Figure 7 shows that the dissipated energy decreases with the increase in the modulus of ductility for bituminous mix based on graphite powder. On the other hand, when the modulus of ductility increases with the addition of steel fiber, the dissipated energy increases.
For bituminous mix based on graphite powder, the energy dissipated decreases when the modulus of ductility increases, under the effect of the intrinsic characteristics and the addition of graphite powder which make the mix more rigid. Consequently, the material becomes more brittle, which will decrease its ductility.

However, with the addition of steel fibers, the ductility modulus increases when the dissipated energy increases, due to the stronger bond between the steel fibers and the bitumen which prevents the development of cracks and microcracks.

### 3.2 Electrical resistivity test result

**Electrical resistivity test of bituminous mix based on steel fibers:** Figure 8 displays the electrical resistivity of the bituminous mixture with various amounts of steel fibers.

The electrical resistivity of the bituminous mix is obtained by a non-destructive test: this is the electrical resistivity test. The effect of steel fiber content on bituminous mix resistivity is illustrated in Figure 8.

The electrical resistivity curve of bituminous mixes based on steel fibers has four phases:
First phase (insulation phase): this is the phase of high resistivity greater than or equal to $1.77 \times 10^9 \ \Omega \cdot m$ with a steel fiber content of 0.8% or less and an electrical resistivity decrease rate of 78.88% or less or the fibers are so separated that there is no path conductor between the two ends of the specimen, here the resistivity of the specimen is similar to that of the resistivity of the control bituminous mix.

Second phase (transition phase): this is the phase where the resistivity ($\Omega \cdot m$) varies between $1.77 \times 10^9$ and $5.06 \times 10^2$, where the percentage of fibers varies between 0.8% and 1% and a rate of decrease in electrical resistivity between 78.88% and 23.07%. The first percolation of conductive paths is formed there and the resistivity drops very rapidly. In this phase, the fibers begin to be in contact.

Third phase (conduction phase): this is the low resistivity phase equal to $5.06 \times 10^2 \ \Omega \cdot m$, with the percentage of steel fibers equal to 1%, and the rate of electrical resistivity decrease equal to 23.07%. In this phase, the fibers reach their maximum level of dispersion in the mixture.

Fourth phase (excess fiber phase): this is the phase with minimum resistivity value less than or equal to $5.06 \times 10^2 \ \Omega \cdot m$, with percentages of steel fibers greater than 1% and a rate of decrease in electrical resistivity greater than or equal to 23.07%; the length of the conductive paths does not decrease and the resistivity decreases slightly with increasing fiber content, once the shortest conductive path is reached, the increase in percentage of conductive fibers does not produce an increase in conductivity.

Electrical resistivity test of bituminous mix based on graphite powder: Figure 9 illustrates how electrical resistivity varies in relation to the amount of graphite powder.

![Figure 9 – Electrical resistivity of the bituminous mix according to the different percentages of graphite.](image)

It is noted that the electrical resistivity decreases with the increase in the graphite powder content. In other words, when the graphite powder increases, the electrical conductivity increases. Graphite slightly lowers the optimum resistivity because if there is enough concentration, its particles tend to make bridges with the graphite powder, thus minimizing the length of the conductive path. Nevertheless, this increase in conductivity has a limit imposed by the minimum distance between the two ends of the specimen studied.

The graphite is randomly distributed in the mixture. Graphite particles form a continuous electrically conductive access when the graphite content reaches a certain level, and the contact resistance between the graphite particles reduces the resistivity.
The addition of graphite fills the gaps in the bituminous mixture. The interior air space of the asphalt mixture rapidly decreases under the external force, and the interior becomes more compact. This increases the contact between the particles and the graphite and forms an electrically conductive access. As a result, the electrical resistance of the specimens will increase, and the electrical resistivity will decrease.

Furthermore, the resistivity of the bituminous mix containing the graphite powder gradually decreases with the increase in graphite content. When the graphite content is increased to 30%, the resistivity of the bituminous coating would have already reached a relatively low level of $2.24 \times 10^7$ (Ω.m) with a rate of decrease in electrical resistivity equal to 62.69%.

A drop in resistivity exists between 20% and 30% graphite, and then, the resistivity gradually decreases with a rate of decrease in electrical resistivity varied between 92.23% and 62.69% with increasing percentage of graphite.

The electrical conductivity of bituminous mixes based on graphite powder increases. So we can distinguish two phases:

- High resistivity phase, with a graphite powder content of less than 20% (insulating behavior).
- Transition phase, between 20% and 30% graphite powder.

### 3.3 Comparison of the bituminous mix's mechanical and electrical properties based on steel fibers

The objective of this work is to make an electrically conductive bituminous coating meeting the electrical requirements without influencing the performance of the mechanical properties.

In order to accomplish this goal, mechanical tests (FENIX direct tensile test) and electrical resistivity tests are used to compare the bituminous mixes' mechanical and electrical properties with steel fibers.

Figure 10 shows the electrical resistivity of the bituminous mix as a function of the energy dissipated for different percentages of steel fibres.

It is noted that the electrical resistivity decreases when the dissipated energy increases. The resistance to cracking increases when the bituminous mix becomes more conductive.

This relation obeys the law of an equation of order 4 with a correlation coefficient $R^2 = 0.83$ (mean correlation).
The electrical resistivity decreases with the increase in dissipated energy because the mechanical bond between the steel fiber and the bituminous binder makes it possible to minimize the microcracks in the asphalt test specimens.

Electrical resistivity decreases with the addition of steel fibers as tensile strength, ductility, and energy dissipated increase. The tensile strength, the dissipated energy, the ductility modulus, and the electrical resistivity of the mix have the best values with the optimum content of steel fibers (1%). Therefore, the bituminous mix based on steel fibers (1%) has better mechanical and electrical properties.

3.4 Comparison of mechanical properties with electrical properties of bituminous mix based on graphite powder

The variation of the electrical resistivity of the bituminous mix as a function of the energy dissipated for different percentages of graphite powder is illustrated in Figure 11.

![Figure 11 - Electrical resistivity of the bituminous mix as a function of the energy dissipated for different percentages of graphite powder.](image)

The law which obeys the electric resistivity according to the dissipated energy is an equation of order 2:

\[
\log \rho = -1.49 \times 10^{-4} (E_d)^2 + 0.057 E_d + 6.11 \quad R^2 = 0.984
\]

We have a very good correlation coefficient, \( R^2 = 0.98 \approx 1 \), so there is a very good correlation between the electrical resistivity and the dissipated energy of the bituminous mix based on steel fibers.

Figure 11 illustrates that the electrical resistivity of the bituminous mix based on graphite powder decreases with the increase in energy dissipated. In other words, when the mix tends towards conductivity, the resistance to cracking becomes higher.

In the percentage (30%) of graphite powder, there are better values of tensile strength and electrical resistivity. Similarly, the crack resistance is higher at a percentage (30%) of graphite powder whose structure causes molecular interactions between the graphite layer structures.

Consequently, the mix containing graphite is prone to producing an infill slip when the asphalt samples are under tensile force. In addition, the graphite powder improves the bond between the bituminous binder and the aggregates. Therefore, when graphite is added, the electrical resistivity decreases so that the tensile strength increases.
3.5 Numerical simulation for determining the conductivity of bituminous mix modified by graphite

In our work, the percentages of graphite powder used in making the bituminous mix are of the order of: (1%, 3%, 5%, 10%, 15%, 20%, 25% and 30%) of graphite powder to volume of bitumen.

In the experimental tests, the maximum percentage (30%) of graphite powder does not reach the conduction phase with an electrical resistivity equal to 2.24.10^{-7} \Omega.m and does not give a conductive bituminous mix. This is why we are going to look for a percentage that transforms the graphite-based bituminous mix into a conductive bituminous mix.

Figure 12 illustrates the correlation curve between the energy dissipated and the percentages of graphite.

Figure 12 shows that there is a very good correlation between the dissipated energy and the percentages of graphite, with a very good correlation coefficient of \( R^2 = 0.98 \approx 1 \).

The law that represents the dissipated energy and the percentages of graphite is an equation of order 2 presented by:

\[
E_d = 603.42(g)^2 + 450.32(g) + 165.92
\]

Where \( E_d \) is the dissipated energy and \( g \): the percentage of graphite powder.

Equation 7’s numerically dissipated energy (\( E_d \)) is obtained by substituting the graphite percentage values.

Figure 13 presents a comparison of the experimentally dissipated energy to the numerically dissipated energy.
Figure 13 – Comparison of the experimental dissipated energy to the numerical one.

Figure 13 confirms that the results of the numerical dissipated energy are identical to the results of the experimental dissipated energy.

In the second step, we replace the values of the dissipated energy found numerically by equation 7 in equation 6 which clearly correlates the electrical resistivity to the dissipated energy (correlation coefficient $R^2 = 0.98 \approx 1$). To find the values of the electrical resistivities at percentages greater than 30% (Table 3).

<table>
<thead>
<tr>
<th>Graphite percentage (%)</th>
<th>Numerical dissipated energy (kN/mm)</th>
<th>Log electrical resistivity (Ω.m)</th>
<th>Electrical resistivity (Ω.m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>165.92</td>
<td>11.50</td>
<td>3.17.10$^{11}$</td>
</tr>
<tr>
<td>1</td>
<td>170.48</td>
<td>11.53</td>
<td>3.42.10$^{11}$</td>
</tr>
<tr>
<td>3</td>
<td>179.97</td>
<td>11.58</td>
<td>3.81.10$^{11}$</td>
</tr>
<tr>
<td>5</td>
<td>189.94</td>
<td>11.60</td>
<td>4.10$^{11}$</td>
</tr>
<tr>
<td>10</td>
<td>216.99</td>
<td>11.51</td>
<td>3.23.10$^{11}$</td>
</tr>
<tr>
<td>15</td>
<td>247.04</td>
<td>11.15</td>
<td>1.41.10$^{11}$</td>
</tr>
<tr>
<td>20</td>
<td>280.12</td>
<td>10.44</td>
<td>2.78.10$^{10}$</td>
</tr>
<tr>
<td>25</td>
<td>316.21</td>
<td>9.30</td>
<td>2.10$^9$</td>
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<td>30</td>
<td>355.32</td>
<td>7.63</td>
<td>4.22.10$^7$</td>
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<td>35</td>
<td>397.45</td>
<td>5.31</td>
<td>2.04.10$^5$</td>
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<td>36</td>
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<td>38</td>
<td>424.18</td>
<td>3.57</td>
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<td>433.32</td>
<td>2.92</td>
<td>8.34.10$^2$</td>
</tr>
<tr>
<td>40</td>
<td>442.60</td>
<td>2.24</td>
<td>1.74.10$^2$</td>
</tr>
<tr>
<td>45</td>
<td>490.76</td>
<td>-1.70</td>
<td>1.99.10$^{-2}$</td>
</tr>
<tr>
<td>50</td>
<td>541.94</td>
<td>-6.65</td>
<td>2.24.10$^{-7}$</td>
</tr>
</tbody>
</table>

Table 3 confirms that the percentage of 39% graphite powder is the percentage that modifies the bituminous mix during the addition of graphite to a conductive bituminous mix. Figure 14 presents the comparison between experimental and numerical electrical resistivity.
Figure 14 – Comparison between experimental and numerical electrical resistivity.

From Figure 14, we confirm the very good correlation between the experimental and numerical results of the electrical resistivity. This figure shows the electrical resistivity of graphite-based bituminous mixes in four phases:

1- **Insulation phase**: it is the phase of high resistivity greater than $2.78 \times 10^{10} \Omega \cdot m$ with a graphite content less than or equal to 20% where the graphite powder particles are so separated that it does not there is no conductive path between the two ends of the specimen, here the resistivity of the specimen is similar to that of the resistivity of the reference bituminous mix.

2- **Transition phase**: this is the phase where the resistivity varies between $2.78 \times 10^{10} \Omega \cdot m$ and $8.34 \times 10^{2} \Omega \cdot m$ with a graphite content varying between 20% and 39% and where the first percolation is formed conductive paths and the resistivity slowly decreases with a gradual transition. In this phase the graphite particles begin to be in contact.

3- **Conduction phase**: this is the resistivity phase less than or equal to $8.34 \times 10^{2} \Omega \cdot m$ and greater than $1.99 \times 10^{-2} \Omega \cdot m$ with the percentage of graphite greater than or equal to 39% and less than 45%, where the graphite particles have reached their maximum level of dispersion in the mixture.

4- **Graphite excess phase**: this is the phase of minimum resistivity value less than or equal to $1.99 \times 10^{-2} \Omega \cdot m$ with percentages of graphite greater than or equal to 45% where there is more graphite; the length of the conductive paths decreases with decreasing resistivity.

4. **Conclusion**

In this work, we present the results of the direct traction test (FENIX test) and the electrical resistivity test carried out on the conductive bituminous mix based on the two additives (steel fiber and graphite powder). The main conclusions are summarized as follows:

- The addition of 1% steel fibers produces a conductive bituminous mix with good performance in terms of crack resistance. It clearly increases the tensile strength, the dissipated energy, and the ductility of the bituminous mix.

- The incorporation of graphite powder at a percentage of 30% improves the conductivity of the mix. This modification by the addition of graphite increases the tensile strength and the energy dissipated, but it has a negative effect on the ductility.
Through a numerical simulation, we have achieved the desired conductivity of the mix with the addition of 39% graphite.

The conductive bituminous mix with steel fibers has good mechanical and electrical performance compared to that with graphite powder.

References


