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Analysis of mortar with brake lining waste by electrical impedance spectroscopy

João B. L. Palma e Silva[1], *Camila T. Ozaki e* Silva[1], *Stephanie C.* Marçula[1], *Ariane R.* Becker[1], *Pedro* Serna[2], *Rosa C. C.* Lintz[1] and *Luisa A.* Gachet[1*]

[1]Universidade Estadual de Campinas (Unicamp), Faculdade de Tecnologia, Limeira-SP, Brazil
[2] Valencia Polytechnic University (UPV), Spain

Abstract. Several researchers have been committed to developing multifunctional mortars, that is, beyond those usual purposes, such as laying masonry, coating, and sealing. These multifunctional mortars may be able to regenerate, store energy, and self-monitor, among other features. Some of these features involve the need to increase the electrical conductivity of the mortar. In this sense, a cement mortar was produced with gradual replacement of the sand with the brake lining waste, to evaluate the electrical impedance and phase angle in a frequency spectrum from 40 Hz to 100 kHz. The specimens had aluminum electrodes embedded in them to measure the properties in question, in the hardened state. This work is a complement to preliminary research that evaluated compressive strength and impedance only at a frequency of 60 Hz, in mortars with the same mix proportion. The results indicated that increasing the content of brake lining waste when replacing sand was able to reduce electrical resistance, both at low and high frequencies. This reduction was due to the increase in electrical conductivity caused by the composition of the brake lining waste, which gives the waste ohmic characteristics. In addition to improving electrical properties, the use of brake lining waste helps to reduce waste disposal in landfills, as well as reducing the consumption of natural aggregates.

1 Introduction

To understand whether a material conducts electricity, electrical resistivity and electrical conductivity must be considered: two inversely proportional electrical properties. Materials with low resistivity and high conductivity are excellent conductors of electricity. Materials with high resistivity and low conductivity are insulators, poor conductors. [1]

Understanding whether a material conducts electricity through resistivity and conductivity are valid for materials that have simple electrical relationships that obey Ohm's Law [1]. However, materials such as cementitious composites are complex systems and cannot be understood through these simple relationships.

* Corresponding author: gachet@unicamp.br

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To this end, cementitious composites have been increasingly analyzed using electrical impedance: an electrical property that considers capacitive and inductive effects that influence the passage of electrical current. [2]

To measure electrical impedance, an alternating current (AC) voltage is applied. By measuring the amplitude and phase of the applied electrical current over a range of frequencies, an impedance spectrum is obtained [3]. From this spectrum it is possible to correlate with conductivity to understand the conductive power of the new material.

It is known that cementitious composites have electrical resistivity values that are not repeated and are small or immeasurable [4]. This electrical resistivity should decrease with the gradual increase in the content of conductive material in the composite [5]. Because electrical resistivity is the inverse of electrical conductivity, it can be said that electrical conductivity increases, becoming measurable. In this way, the cementitious composite that had usual purposes (laying masonry, coating, and sealing), may have properties of self-healing, electromagnetic shielding, store energy, and self-monitor, among other features (Fig. 1).

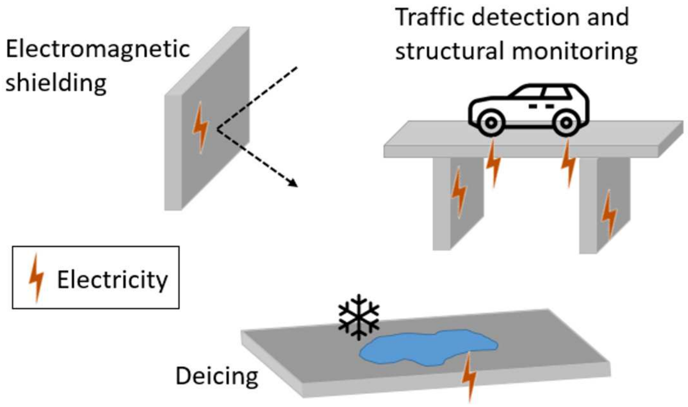

Fig. 1. Applications of cement composites with electrical properties.

Researchers have reported over the last few years the application of cement composites with electrical properties, for example, for the production of self-sensing bricks for monitoring masonry structures [6]. Other research presents its use in detection and monitoring systems for vehicles on roads [7] and even on railway ties [8].

A lot of research has been carried out on the addition of carbon-based materials to cementitious composites [4-10]. However, these materials have a high cost and, because they are noble, such as carbon nanotube, graphene, and nickel powder, they increase environmental impacts. Seeking sustainability, authors have researched the use of waste or recycled materials [11].

Wang et al. (2017) [12] investigated mortars with break-shoe waste replacing fine aggregate in proportions of 0 (reference), 5, 10, 15, 20 and 30% and a water/cement ratio of 0.43. They found that the mortar with waste break-shoe continues to have good compressive strength when compared to the reference [12]. This is a good sign, as it demonstrates that compressive strength may not be significantly affected using waste materials in the composition of the cementitious composite.

The aim of this work is analysis of a mortar with brake lining waste as conductive materials. This work is a complement to preliminary research [13] that evaluated compressive strength and impedance only at a frequency of 60 Hz. The same specimens, but with different ages, were used use in both papers. In this work electrical impedance, phase angle and conductivity were investigated on a frequency spectrum (40 Hz – 100 kHz) to understand this new sustainable material.

2 Methodology

The mix was adapted from the literature [12] with a mass ratio of 1:1 (cement and fine aggregate) and a water-cement ratio of 0.37. The fine aggregate was replaced by brake lining waste at 0 (reference), 5, 15, 40, 50, 60 and 70% [13]. This research was carried out at the Construction Materials Laboratory of the Unicamp Faculty of Technology (FT-Unicamp).

2.1 Materials

The cement used was Holcim's Portland Cement, type CP-V (grade >34 MPa at 7 days), a high early strength comparable to ASTM Type III cement. The fine aggregate used was quartz sand, with maximum diameter of 2.4 mm and a Fineness Modulus of 2.31.

The brake linings used were from heavy vehicles (trucks), which were obtained from a mechanical workshop in Campinas-SP/Brazil (Fig. 2a). The brake linings were chosen based on their electrical resistance (<5.0 kΩ), measured between the two multimeter probes, at a distance of ≈ 10.0 mm. After obtained, the brake linings went through a crushing and grinding process until they had maximum diameter of 6.3 mm and a Fineness Modulus of 3.02 (Fig. 2b) according to the procedure in Silva et al. (2024) [13].

Fig. 2. Used brake lining (a) and brake lining waste grinded (b).

2.2 Methods

Table 1 shows the mix proportions and nomenclature adopted in this work. Although there was a reduction in the workability of the mortar, no superplasticizer was used.

Table 1. Mix proportions of mortar.

Identification	Cement (kg/m³)	Sand (kg/m³)	Brake lining waste (kg/m³)	Water (kg/m³)
TA-00	935,87	935,87	-	346,27
TA-05	935,87	889,08	35,01	346,27
TA-15	935,87	795,49	105,02	346,27
TA-40	935,87	561,52	280,06	346,27
TA-50	935,87	467,94	350,07	346,27
TA-60	935,87	374,35	420,09	346,27
TA-70	935,87	280,76	490,10	346,27

The homogenization of the mortar was carried out in accordance with NBR 16541:2016 [14], similar to UNE-EN 1015–3. Cubic specimens (40 mm) were molded according to NBR 16868-2:2020 [15], similar to UNE-EN 196–1. Aluminum electrodes with an area of 700 mm² (20 x 35 mm) were embedded, leaving 200 mm² protruding from the mortar (Fig. 3). The specimens were cured for 28 days in a humid chamber (18 ºC and 84% relative humidity).

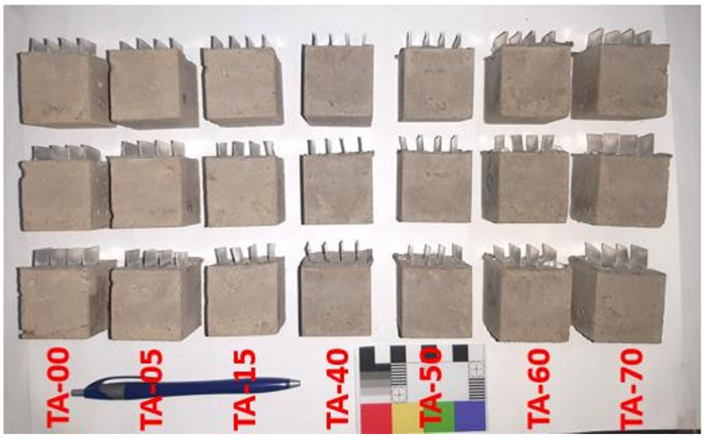

Fig. 3. Cubic mortar specimens with electrodes.

The system used for electrical impedance spectroscopy is shown in figure 4. Impedance analyzer was an Agilent model 4294A equipment. The measurement frequency range was from 40 Hz to 1 MHz at an amplitude of 1.00 V. The electrical impedance (Z) and phase angle (θ) were measured, in specimens aged >28 days. From these measurements, it was possible to determine the electrical resistance (R), resistivity (ρ), electrical conductivity (σ), imaginary impedance (Z”), according to equations 1 to 4.

$$R = Z * cos\ (\theta) \quad (1)$$
$$\rho = R * (A/d) \quad (2)$$
$$\sigma = 1 / \rho \quad (3)$$
$$Z'' = Z * sen\ (\theta) \quad (4)$$

In equation 2, *A* is the area (500 mm²) of the part of the electrode embedded in the mortar and *d* is the distance (10 mm) between the electrodes.

With these measurements, it was possible to plot graphs of electrical conductivity x frequency, imaginary impedance x electrical resistance (Nyquist plot) and phase angle and electrical impedance x frequency (Bode plot).

Analysis of variance (one-way ANOVA) was performed, followed by a multiple comparison test (Tukey) of conductivity in relation to brake lining waste content at frequencies of 40 Hz, 40.89 Hz and 100 kHz. This was done to identify whether there is a significant difference at the 95% level.

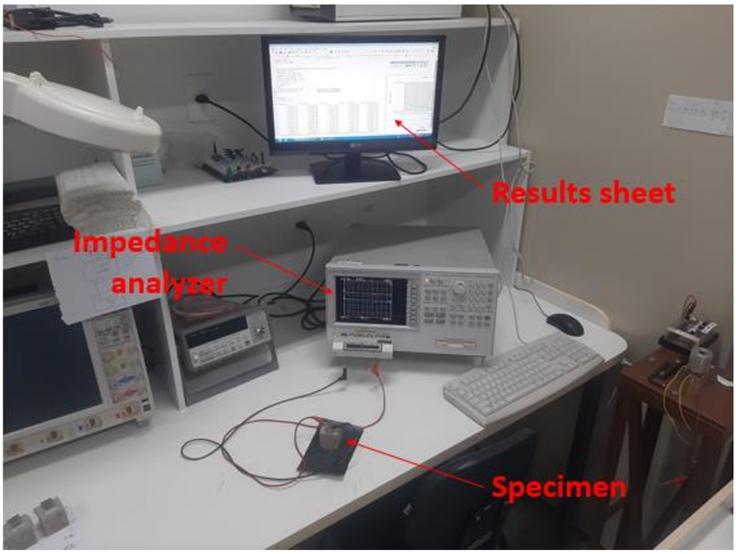

Fig. 4. Impedance and phase angle measurements on the mortar specimen.

3 Results and Discussions

In the electrical conductivity analysis (Fig. 5) it is possible to observe that the brake lining waste contributed to its increase. However, at low frequencies (up to ≈ 2000 Hz) the reference mix proportion (TA-00) showed greater conductivity than the TA-15. This situation may be associated with the fact that at low frequencies, the electrode resistance prevails over that of the mortar [16].

In a segmented analysis at frequencies (Fig. 6) of 40 Hz, 49.89 kHz, and 100 kHz, it was found that at the lowest frequency analyzed (40 Hz), there was no increase in electrical conductivity. The increase is evident from the TA-40 mix proportion in the average (49.89 kHz) and maximum (100 kHz) frequencies analyzed.

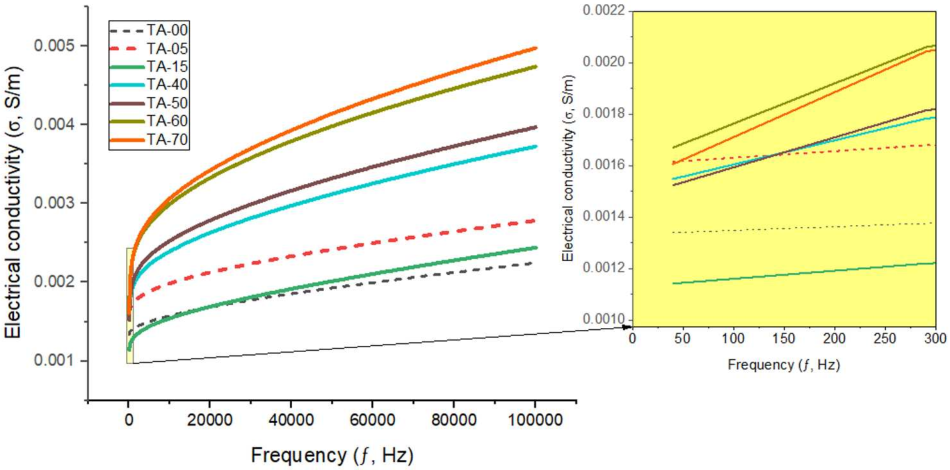

Fig. 5. Average electrical conductivity from 40 Hz to 100 kHz in the mortar specimens.

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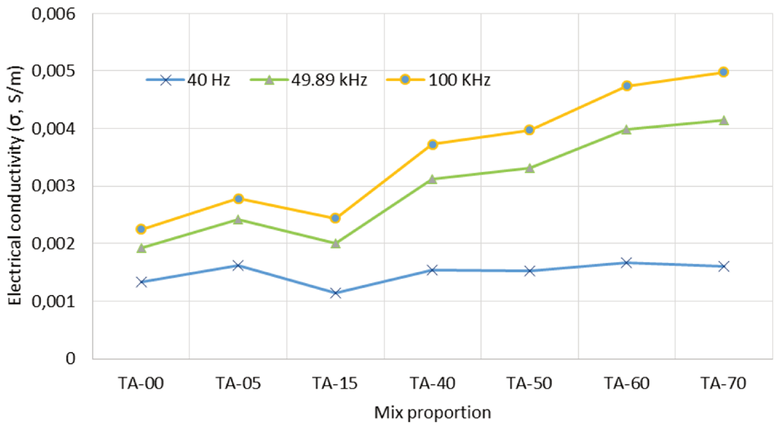

Fig. 6. Average electrical conductivity in 40 Hz, 49.89 kHz and 100 kHz in the mortar specimens.

Still in the analysis segmented by frequencies, in figure 7, it is possible to observe that through graphic interpolation, the electrical conductivity starts at 0.0025 S/m at frequencies between 40 Hz and 49.89 kHz.

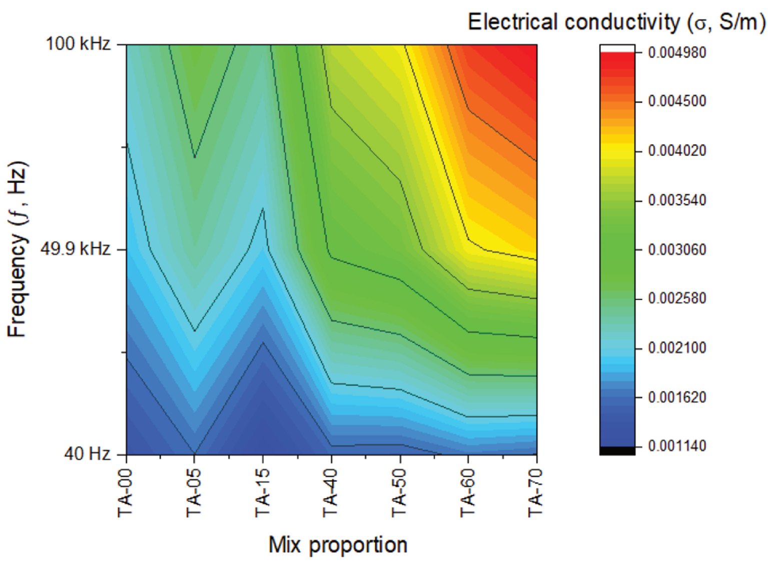

Fig. 7. Average electrical conductivity in 40 Hz, 49.89 kHz and 100 kHz in the mortar specimens.

Through variance analysis (one way ANOVA) at 40 Hz, there was no significant increase in electrical conductivity between the samples, as P-value (0.38898) was greater than the critical significance level (0.05). In the cases of variance analyses at 49.89 kHz and 100 kHz, the P-value was <0.0001, that is, it confirmed the hypothesis of a significant difference between the results.

As already indicated by the analysis of variance at 40 Hz, the results of the Tukey test at the same frequency confirmed that there was no statistically significant difference between the average conductivities, as shown in figure 8.

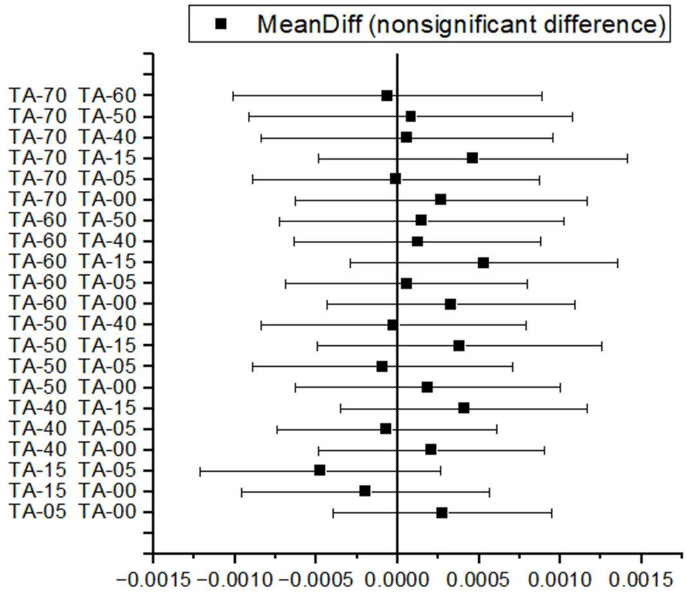

Fig. 8. Tukey test of electrical conductivity (S/m) in 40 Hz in the mortar specimens.

In the case of the Tukey test analysis for the average results at 49.89 kHz, it was found (Fig. 9) that at mix proportion TA-40, there was a statistically significant increase in electrical conductivity in relation to the reference mix proportion (TA-00). In the analysis at 100 kHz, the statistically significant increase occurred only from the mix proportion TA-50, as shown in figure 10.

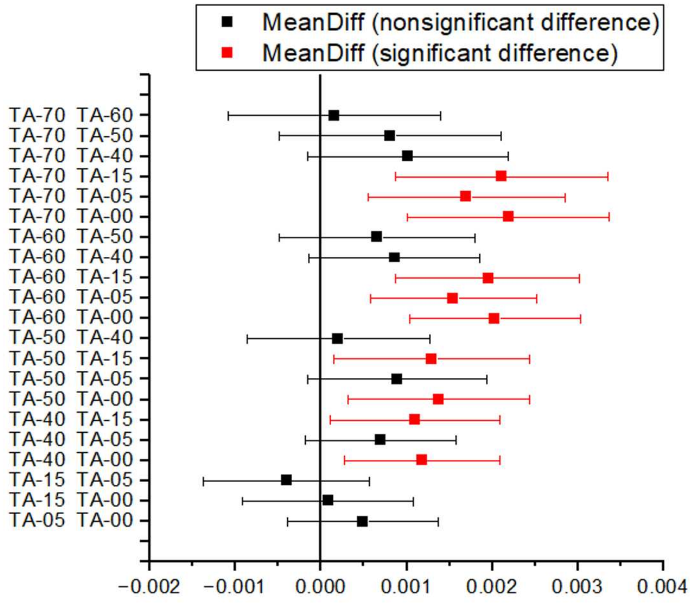

Fig. 9. Tukey test of electrical conductivity (S/m) in 49.89 kHz in the mortar specimens.

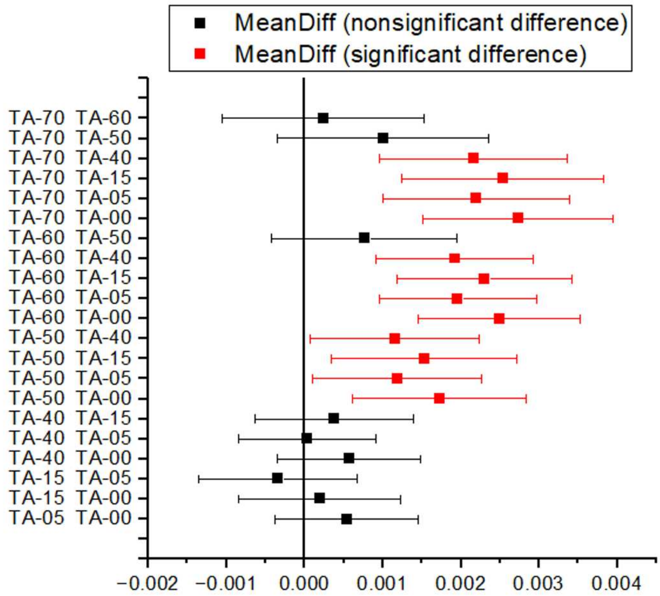

Fig. 10. Tukey test of electrical conductivity (S/m) in 100 kHz in the mortar specimens.

Using the Nyquist plot (Fig. 11) it is possible to observe the approximate frequency of the cusp point, which may represent the transition point between the resistance of the electrode and the mortar [16]. Still in the Nyquist plot, it is observed that from mix proportion TA-40, there was less influence of reactance on electrical resistance.

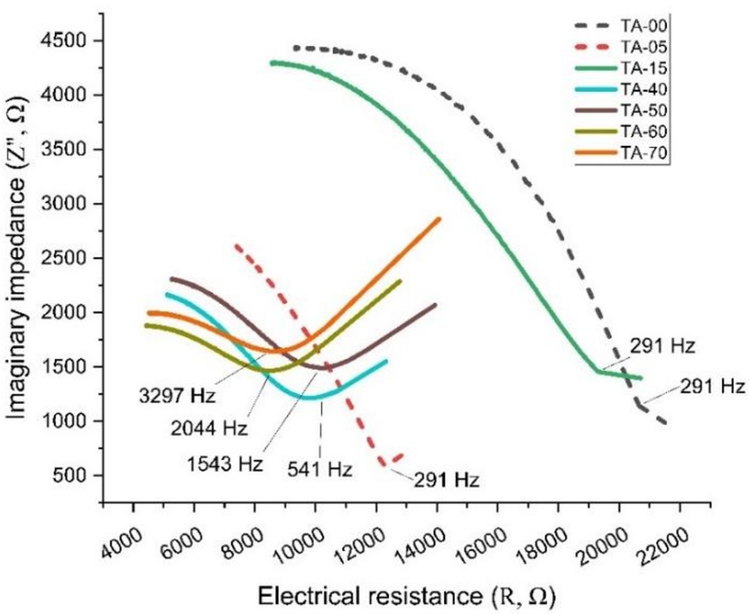

Fig. 11. Nyquist plot analysis in the mortar specimens.

As the frequency increases, the impedance and phase angle change (Fig. 12), which shows that the mortar does not have a purely resistive behavior, which was already expected.

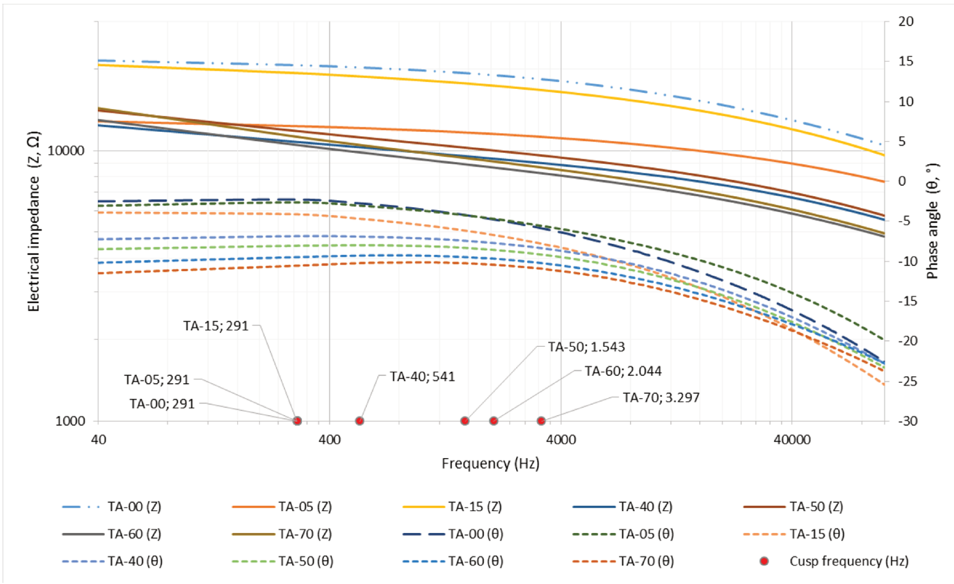

Fig. 12. Bode plot analysis in the mortar specimens.

The Bode plot also showed that with the increase in brake lining waste in the mortar, it presented a reduction in its electrical impedance, which was associated with the change in phase angle and directly reflected in the increase in electrical conductivity.

4 Conclusions

This work aimed to evaluate the electrical properties of mortar with brake lining waste using impedance spectroscopy, with the aim of collaborating with future applications aimed at using electrically conductive mortars.

Based on the results obtained, it is possible to conclude that:

1. There was an increase in the electrical conductivity of the mortars through the gradual replacement of sand with brake lining waste, at different frequencies analyzed;
2. The statistically significant increase in the electrical conductivity of the TA-40 mix proportion, especially at the frequency of 49.89 kHz and higher;
3. With the incorporation of the waste, the mortar had less influence on the capacitive reactance properties of the electrical resistance;
4. The use of brake lining waste in the production of mortars with electrical properties can contribute to reducing environmental impacts related to the final disposal of these wastes in the environment.

The satisfactory results obtained in this work, as well as in the evaluation of the mechanical properties of these same samples [13], demonstrate that mortars with brake lining waste have potential for further investigations regarding their physical-mechanical, electrical, and environmental properties.

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