

Comparison of the mechanical and dielectric characteristics of various polymers mixed with Ground Tired Rubber (GTR) for its application as industrial work footwear insulator

Ramon Mujal-Rosas¹, Marc Marin-Genesca², Jordi Garcia-Amoros³, Xavier Colom-Fajula¹ y Xavier Salueña-Berna¹

¹ Universidad Politécnica de Cataluña. ESEIAAT. Colom, 1 - 08222 Terrassa, Barcelona (España).

² Universidad Rovira i Virgili. ETSEQ. Avenida Països Catalans, 26 - 43007 Tarragona (España).

³ Universidad Rovira i Virgili. ETSE. Avenida Països Catalans, 26 - 43007 Tarragona (España).

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ABSTRACT:

The massive manufacture of tires and their difficulty for subsequent recycling, represents today, a serious environmental problem. There are several methods to try to recycle used tires, one of them is mechanical separation, in which the vulcanized rubber is separated from steel and fibers, which results in a rubber that can be milled into small particles (Ground Tire Rubber, or GTR). This rubber can be used in various applications, such as the one presented by this research, in which, once mixed with customary polymers, it should be used for the manufacture of insulation for industrial work shoes. Therefore, the objective of this study is to evaluate the results of mixing these GTR particles with several commonly used polymer matrices (polymer + GTR), verifying that both their mechanical and dielectric properties comply with the standards established for the manufacture of the insulation for shoes for industrial use. The analysis will be carried out using seven polymer matrices mixed with different concentrations of GTR.

Keywords: GTR, Recycling, Electrical properties, Mechanical properties, Insulation, Use as isolation of work shoes.

1. - INTRODUCTION

The global problem of the accumulation of used tires [1-2] has driven the efforts of the scientific community to find solutions that will lead to the recovery and reuse of this wasted material. The use of this Ground Tire Rubber (GTR) as reinforcements in composite materials has been extensively studied in several research papers [3]. The presence of GTR in polymer matrix composite materials modifies both the dielectric and mechanical behavior of the materials. In general, a thermoplastic or thermosetting polymer acts as a matrix, while the elastomer, or GTR, acts as a dispersed phase [4-6]. As in other mixtures of two-phase polymers [7-8], the expected interfacial compatibility between the components is important in order to achieve the appropriate properties for later industrial use. In the case of recycled elastomers, the expected compatibility is low, so it is necessary, to increase this compatibility, to reduce the degree of cross-linking of the GTR by proper devulcanization [9-11]. Significant changes in mechanical or dielectric properties are also observed, when we vary the size of the reinforcing particles (GTR) [12].

In previous research [13], it has been shown that the best mechanical and dielectric properties are obtained with GTR particle diameters below 200µm, worsening with average particle sizes 200 <p <500 µm, and being significantly lower, with large particles p > 500µm. Therefore, this research will focus on GTR particles whose diameter is less than 200µm. In principle, it is based on the hypothesis that the mechanical and dielectric properties of the polymer matrix will worsen when mixed with GTR, being the objective of this investigation, to see how much percentage of GTR can be added to these polymeric matrices (Polyvinylchloride PVC, acetate of ethylene vinyl EVA, high density polyethylene HDPE, polypropylene PP, polyamide PA6, acrylonitrile butadiene styrene ABS and polystyrene PS), keeping the mechanical and dielectric properties within an acceptable range of values, and also maintaining the initial microstructure of the polymer [14]. Finally, it will be indicated if industrial applications are possible for this polymer-GTR mixture, bearing in mind that GTR constitutes the reinforcing agent in these mixtures, so this GTR will not work in applications with high mechanical or dielectric requirements, which if which have their own pure polymers. Most polymeric materials have very good insulating properties, with conductivity values below 10-12 S·cm⁻¹. It is known that with the incorporation of conductive additives such as graphite, carbon black, metal oxides, etc., the conductivity improves significantly and therefore, its insulating capacity worsens. Without a doubt, carbon black is next to glass fiber, the most common and easy to handle additive [15], and therefore, these mixtures represent heterogeneous materials in which the properties depend on the quantity, size and shape of the reinforcement, as well as other factors such as preparation, compatibility, etc. Other researchers [16-17] have studied different samples of materials with different compositions and varying proportions of additives such as carbon black (CB) reaching similar results to those of this research.

1.1. Featured Application

To meet the proposed dual objective: to verify the possibility of obtaining an industrial application from these mixtures, and if so, to determine the maximum percentage of GTR that can be included in the polymer matrix, in this investigation, all the characteristics were characterized. samples both mechanically and dielectrically and from the values obtained, the possible application to the use of insulating soles of industrial footwear was determined. The necessary parameters set by the standards for this use are: conductivity $>10^{-6}$ S/cm, tensile strength >10 -12MPa and elongation at break $>450\%$, according to the regulations: UNE-EN ISO 20345/6/7:2005 and UNE 53510.

2. MATERIALS AND METHODS

2.1. Materials

The polymers used for this study have been: polyvinyl chloride (PVC); High density polyethylene (HDPE) recycled from water bottles; Copolymer of ethylene vinyl acetate (EVA), 18% vinyl acetate and 82% ethylene; Polypropylene (PP); Acrylonitrile butadiene styrene (ABS) composed of 30% acrylonitrile, 20% butadiene and 50% styrene; Polyamide 6 (PA6), known as Nylon 6; Polystyrene (PS); and the recycled tires (GTR), with a particle size of less than $700\mu\text{m}$, verifying by means of TGA analysis that their carbon black content is of the order of 35%. The original GTR was screened into three categories of particle size: $p < 200\mu\text{m}$, $200 < p < 500\mu\text{m}$ and $p > 500\mu\text{m}$, using only particles smaller than $p < 200\mu\text{m}$ for this article. In table 1, several properties of these polymers are detailed.

2.2. Compound Preparation

The GTR recycled tire powder was dried in an oven at 100°C for 24 hours. A total of seven samples were prepared for each polymer, six of the Polymer/GTR compound, varying the composition of GTR (5%, 10%, 20%, 40%, 50% and 70% GTR), plus one sample of each polymer Pure with the same requirements. The mixing process was carried out with a Brabender Mixing machine (Fig. 2.a), at different temperatures, and to avoid polymer degradation the mixing time was limited to 4min. Polymer / GTR laminates were obtained by using a hot plate press at 100kN and different temperatures (Table 1) for about 10min, depending on the polymer to be treated. The cooling stage was performed with a closed water circuit, which was maintained at the same pressure for 5min. The samples for the test were prepared in accordance with the specifications of ASTM-D-638 Type V. Each test was repeated 5 times, discarding the defective values and giving as final value, the average value.

Polymer	Commercial Name	Fluency Index (g/min)	Density (kg/m ³)	Processing Temperature (°C)	Pressing Temperature (°C)
PVC	Etinox	1.35	1.225	195-200 °C	210 °C
EVA	Alcudia PA6 539 type	0.20	937	105-110 °C	120 °C
HDPE	Alcudia 4810-B	1.35	960	150-155 °C	170 °C
PA 6	Ultramid B3S	1.55	1130	195-200 °C	210 °C
ABS	Terluran® HH-106	1.45	1050	180-185 °C	195 °C
PP	Isplen® 099 K2M type	0.55	902	155-165 °C	165 °C
PS	Polystyrol 486 M	1.45	1050	180-185 °C	195 °C

Table 1. Commercial name, fluidity index, density, processing temperatures with Brabender mixer and constant pressure pressing temperatures of 200 Bar, for each material

2.3. Mechanical analysis

The Tension-Deformation tests were performed with a universal Instron 3366-10 kN machine, following the specifications of ASTM-D-638 Type V Standard. The test speed was 20mm/min. The test temperature was $23\pm 2^\circ\text{C}$, and the relative humidity was $50\pm 5\%$. The study of the mechanical properties, according to the concentration of GTR in the matrix and the particle size $p < 200\mu\text{m}$ of GTR, include Young's Modulus, Tractive Strength, Elongation at Break and Tenacity or Hardness. Five samples were used for each test. The mean and standard deviation were calculated for each magnitude, leaving out samples with a defective result.

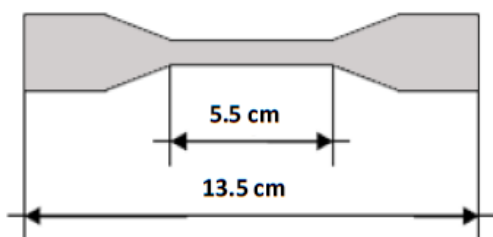
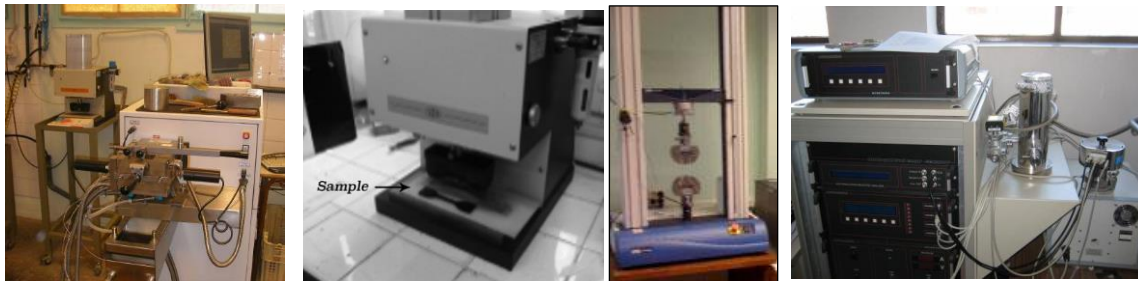


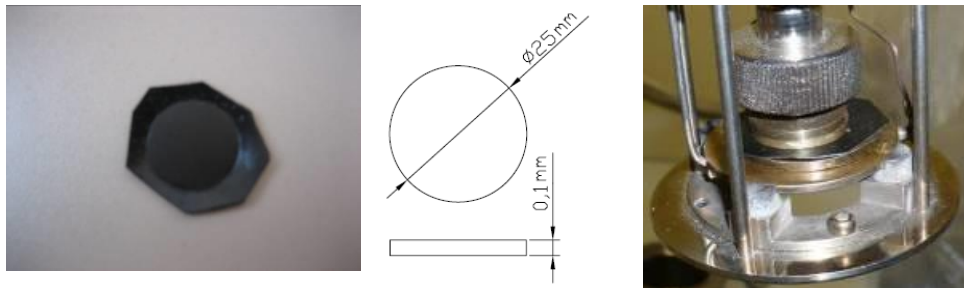
Figure 1. Detail of the samples that were used for the mechanical tests.

2.4. Dynamic electrical analysis

Dielectric analysis was carried out with particles smaller than 200 μm [17]. The dielectric parameters were measured by means of Dynamic Electrical Analysis (DEA) with a BDS 40 broadband dielectric spectrometer, which incorporates a Novoterm Novocontrol temperature sensor and uses compression molds of 2 cm in diameter. The data was obtained using a parallel plate sensor. For this test, a sample was placed between the two electrodes. The dielectric permittivity (ϵ'), the dielectric losses (ϵ''), thus obtaining the Loss Tangent ($\text{Tang } \delta$) as well as the conductivity (C) were measured by means of the DEA (Fig. 2.d). With the defined parameters, the machine was activated giving an alternating current between the two electrodes, which allows to obtain two responses for this current: a resistive intensity and a reactive intensity. The phase difference between these two intensities allows the calculation of the electrical parameters: real permittivity, imaginary permittivity, phase angle and conductivity. The dimensions of the samples are defined by ASTM D-150, the cylindrical samples being 2.5mm in diameter and 0.1mm thick (Fig. 3.a and Fig. 3.b). After obtaining the results of the different samples, the dielectric potential of each mixture was analyzed in depth.



a) b) c) d)
Figure 2. a) Brabender mixing machine, b) Puncher c) Traction-Deformation Test, d) DEA Test



a) b) c)
Figure 3. a) Sample for the DEA test, b) DEA sample dimensions, c) DEA electrodes

3. RESULTS

3.1 Mechanical properties Polymers / GTR

As can be deduced from the analysis of figures (4.a, 4.b) and (5.a, 5.b), the addition of used tire particles generally produces, and in all compounds, a decrease in the four mechanical properties analyzed. Young's Modulus (Fig. 4.a) Tensile Strength (Fig. 4.b), Elongation at Break (Fig. 5.a) and Tenacity or Hardness (Fig. 5.b). If we analyze the Young Module, we can see how PVC and EVA compounds have a better behavior at the beginning with the addition of GTR particles (up to 10%), while for tensile strength it is observed that the compounds of PA6 and PVC show better behaviors with the addition of large amounts of GTR (50% -70%).

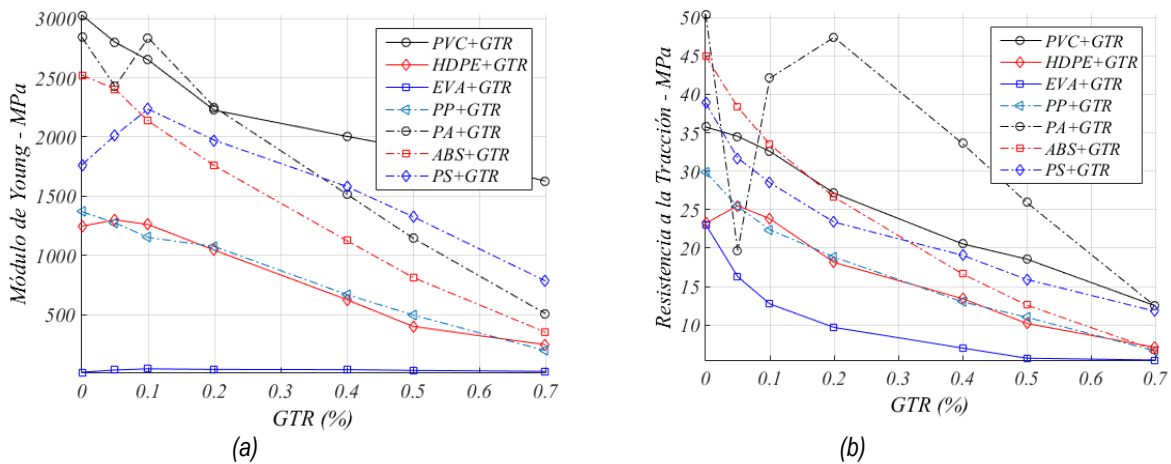


Figure 4. a) Young's modulus (MPa), b) Tensile strength (MPa), for the seven concentrations of the Polymer / GTR compounds and particle size $p < 200\mu\text{m}$.

Thus, for the Young Module (figure 4.a) it can be seen that the materials analyzed have similar characteristics. The PVC, PA6 and ABS (3028.89-2841.47-2522.37 MPa respectively) stand out with a higher Young Module, with the PVC polymer reaching 3028.89 MPa, and secondly, in a range of similar values, the polymer is found PA6 (2841.47 MPa). With the addition of GTR particles, in general, Young's Module decreases. Comparatively, the polymer that exhibits the best properties of Young's Module is PVC, which shows good performance up to concentrations in GTR of the order of 20% (2225.87MPa), however, for percentages of GTR greater than 20% (40 % -50% -70%) the values are considerably reduced (2005.87-1900-1627MPa respectively), these values being 40% of the value of the pure polymer. For the rest of the materials, in general, good behaviors of the Young Module are obtained, so for the PS and the HDPE the addition of GTR positively affects this property, slightly increasing the Young Module for GTR percentages of 5-10 -twenty%. A very prominent case is the pure EVA copolymer, which has very low Young Module properties (13.26MPa) and improves with the incorporation of GTR (41.67 MPa for EVA + 10% GTR), thus presenting some advantages polymer regarding the Young Module with respect to the rest of the compounds analyzed.

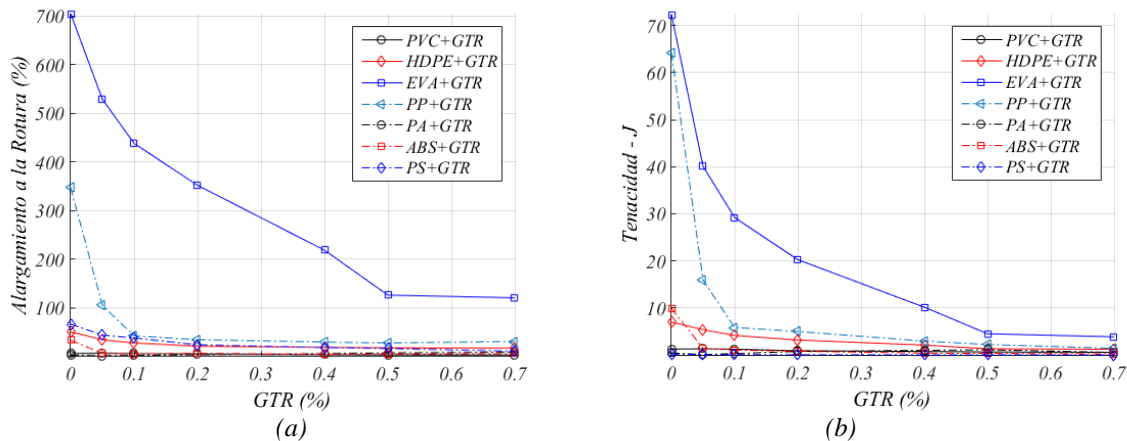



Figure 4. a) Young's Modulus (MPa), b) Tensile Strength (MPa), for the seven concentrations of the Polymer/GTR compounds and particle size $p < 200\mu\text{m}$.

Regarding tensile strength (Fig. 4.b) it can be deduced that the materials analyzed have comparable characteristics in terms of this magnitude. Of all of them, those with the highest tensile strength are PA6, ABS, PS and PVC. Highlighting the PA6 polymer that reaches 50.41MPa, and secondly the ABS polymer (44.98MPa). If we add 5% of GTR particles the tensile strength of PA6 decreases drastically to values of 19.67MPa, to recover quickly up to 42MPa, with 10% in GTR, this is an exception, since in general with the addition of Small percentages of GTR additive (5%) compounds have decreased their tensile strength. Comparatively, the polymer with the best tensile strength properties is PA6 with GTR percentages of up to 20% (47.39MPa), however, for GTR percentages greater than 20% (40% -50% -70 %) the tensile strength behavior falls to half of the values that the polymer would have without additive. For

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the rest of the materials, it can be deduced that, in general, they have good tensile strength behaviors, although for PS and EVA (38.89MPa-23.08MPa respectively) the addition of GTR negatively affects this property, and they suffer a certain drop for percentages greater than 20% of GTR (40% -50% -70%); For example, for PS + 40/50/70% GTR the tensile strength values are: 19.07-15.93-11.80 MPa. For the compound EVA + 40/50/70% GTR the tensile strength values are 7.02-5.70-5.46 MPa, which already represent less than 50% of the value of the pure polymeric EVA matrix.

In general, if we analyze the elongation at break graph (Fig. 5.a), the materials that stand out are the EVA copolymer (704.6%) and in second place is the PP polymer (346.71%), although when particles are added of GTR, the elongation properties of the PP decrease dramatically. Comparatively, the polymer with better elongation properties is therefore EVA, which with percentages of GTR of up to 10-20%, has elongation values of EVA + 10% GTR: 438%; EVA + 20% GTR: 351.53%, however, for the PP and for the same percentages in GTR (10% -20%) its elongation behavior is very poor, reaching only 15% of the polymer without reinforcement (PP + 10% GTR: 41.73%; PP + 20% GTR: 33.89%). Considering the rest of the materials, PS and HDPE have certain elongation properties, although they do not reach 100% elongation at break (PS: 66.27% - HDPE: 50.12%), worsening when adding GTR in percentages of 20% (PA + 20% GTR: 23.30% - HDPE + 20% GTR: 20.01%), which already represent percentages below 50% of the matrix of the pure polymer. It is also observed that the best behavior is shown by EVA when GTR particles are added [18-19], specifically EVA compounds, range from 704%, 528%, and 437%, which means decreases of 29%, 43% and 50% for GTR concentrations of 5%, 10% and 20% respectively. From 40% of GTR concentration, the values are always less than 3.5 times those shown for pure EVA. Thus, it is observed that the reduction in the deformability of the elastomer influences the decrease in elongation at break, and that the decrease in elongation at break is related to the imperfect interfacial adhesion between the components. The rest of polymers have very low values for this magnitude. The incidence of poor adhesion between phases is thus a particularly important result [20-21].

Finally, if we look at the Tenacity or Hardness (J) graph (figure 5.b), it can be concluded that the materials analyzed have very heterogeneous characteristics regarding this property. Thus, for example, the hardness has values of 72.3J for pure EVA and 40J or 29.2J for GTR concentrations of 5% and 10% respectively. For concentrations above 20%, the considerations on the percentage of GTR added are not important, since in any case the energy 10J is not even reached, which means that these values are seven times lower than the original EVA. Of all the compounds analyzed that have a greater hardness or toughness are EVA, and PP, and in a second level the ABS and HDPE polymers would be located. The EVA polymer stands out with values of 72.32J and, secondly, the PP polymer (64.22 J) is in a very similar range of values. With the addition of GTR particles, the hardness properties (J) decrease dramatically. Comparatively, the polymer with the best breaking energy properties is the EVA copolymer, which with GTR percentages of up to 20%, shows a good performance (20.24J), instead for GTR percentages greater than 20% (40% -50% -70%) the energy behavior falls dramatically (10.16J – 4.51J – 3.86J) with values close to 40% of the value of the polymer without additive. For PP, ABS and, to a lesser extent, HDPE, the addition of GTR very negatively affects the properties of the energy at break (J). For HDPE, the addition of GTR does not so severely affect the values of the energy at break. Prominent cases are PVC, PA6 and PS polymers, which have very low values of energy at breakage, making these values worse with the addition of GTR.

3.2. Dielectric Properties Polymers/GTR

3.2.1. Conductivity and loss tangent comparison (Tg δ)

For compounds with GTR (at 30°C), PA6 samples are the most conductive of those analyzed (Figure 6.a). When the GTR content is increased, the conductive behavior of the samples, in most cases, increases up to two orders of magnitude with respect to pure PA6. From 0-5% in GTR, the conductive behavior of the samples is more heterogeneous, with pure PS and PP being the least conductive, at 50 Hz, with conductivities of the order of $1 \cdot 10^{-13}$ and $1 \cdot 10^{-11}$ S/cm respectively. For concentrations in GTR of 70%, PS is shown as the most insulating material ($1 \cdot 10^{-14}$ S/cm) followed by ABS, which for an industrial frequency of 50 Hz shows a conductivity of the order of $1 \cdot 10^{-10}$ S/cm. The most resistive compounds are HDPE, PS and PP, followed by EVA, PVC, and ABS and, finally, PA6 is found, which turns out to be the material with the highest conductivity. Thus, it is observed that the addition of the GTR causes an increase in the conductive behavior of almost all polymers, which tends to converge, except in the case of PA6, which remains two orders of magnitude above the rest of the Polymers analyzed, that is, for a GTR concentration of 40-50%, PA6 shows a conductivity between $4.95 \cdot 10^{-13}$ and $1.73 \cdot 10^{-12}$ S/cm, while for the rest of polymers the conductivity is maintained at around $1 \cdot 10^{-15}$ S/cm, and in the case of ABS and PS, and for concentrations in GTR of 40%, conductivities of between $1.05 \cdot 10^{-14}$ and $1.32 \cdot 10^{-14}$ S/cm are reached, respectively, which indicates a poor dielectric behavior of the compounds, results already obtained in other investigations carried out with carbon black (CB) [22-23]. If we analyze the Tg δ, for the seven Polymer / GTR compounds (Figure 6.b), the material with the highest delta tangent (Tg δ) is polyamide, which without GTR additives has values of $1 \cdot 10^{-1}$, deducing that the addition of GTR affects Tg δ in this case, with increases of an order of magnitude. Materials with lower Tg δ are PP, HDPE, EVA and PS (0.001), followed by PVC and ABS (0.01), and finally, with a higher Tg δ, PA6 (0.1) is located. With the addition of GTR in the matrix, this relationship changes, tending to converge all polymers with a Tg δ of the order of $1 \cdot 10^{-1}$ showing few variations, with the exception of the

compounds formed by GTR with PVC and PA6. For low concentrations of GTR, $T_g \delta$ is heterogeneous and, as GTR is added, the behavior tends to assimilate. In general, $T_g \delta$ ranges from $1 \cdot 10^{-1}$ to $1 \cdot 10^{-2}$ for almost all polymers, except for PA6, which, with the addition of GTR increases $T_g \delta$ by an order of magnitude.

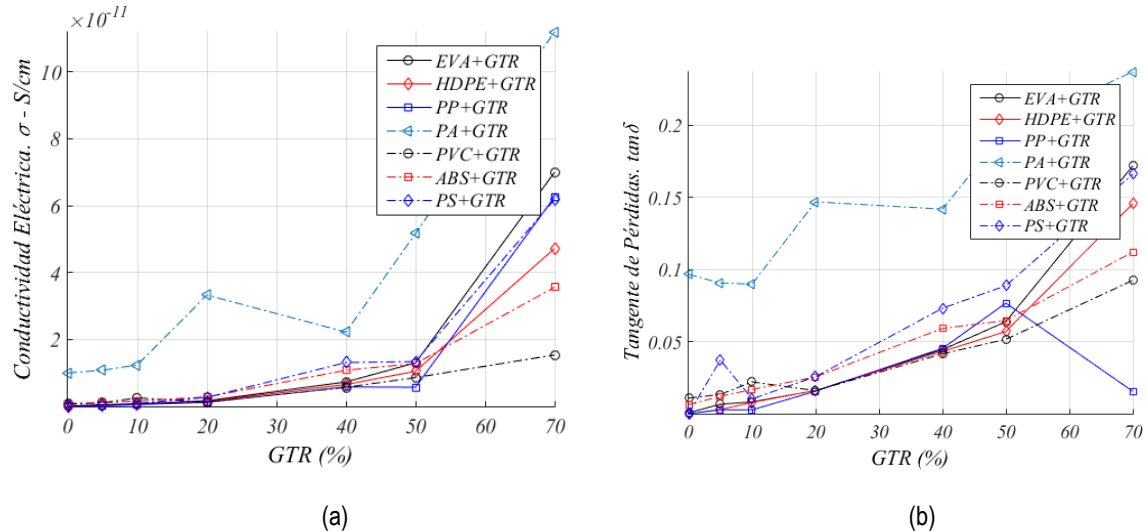


Figure 6. a) Electrical conductivity (σ - S/cm), b) Loss Tangent ($Tan \delta$), for the different concentrations of Polymers/GTR, particle size $p < 200 \mu m$, frequency set at 50 Hz and $30^\circ C$.


4. DISCUSSION

As can be seen in Table 2, the highest values of the analyzed mechanical properties correspond to the pure polymer (0% GTR), with some important exceptions as highlighted in the table. Thus, in the case of EVA, HDPE, PA6 and PS some of these mechanical properties improve with the addition of microparticles ($p < 200 \mu m$) of GTR in the polymer matrix. The mechanical parameters are significantly influenced by the GTR. Basically, it is observed that, at a mechanical level, the properties of the polymer's pure matrix are maintained, approximately, with concentrations of between 10 and 20% of GTR particles. The interaction of the matrix with the GTR particles is quite low and, therefore, the matrix accepts this low GTR content. For values greater than 20% of GTR in the polymer matrix, the mechanical properties decrease drastically in all cases.

Polymer composite	Young's Modulus (MPa)	Tensile Strength (MPa)	Elongation at Break (%)	Tenacity (J)
PVC/GTR	3028.89 (0%GTR)	35.75 (0%GTR)	6.31 (5%GTR)	1.31(5%GTR)
EVA/GTR	41.67 (10% GTR)	23.08 (0%GTR)	704.6 (0%GTR)	72.32(0%GTR)
HDPE/GTR	1300.11 (5%GTR)	25.51 (5%GTR)	50 (0%GTR)	7(0%GTR)
PP/GTR	1368.65 (0%GTR)	29.9 (0%GTR)	346.71 (0%GTR)	64.22(0%GTR)
ABS/GTR	2522.37 (0%GTR)	44.98(0%GTR)	32.91(0%GTR)	9.9(0%GTR)
PA6/GTR	2841.47(0%GTR)	50.41(0%GTR)	8.46(70%GTR)	1.09(50%GTR)
PS/GTR	2235.42(10%GTR)	38.89(0%GTR)	66.27(0%GTR)	0.20(0%GTR)

Table 2. Highest values reached for each property analyzed and the compound considered

As for the dielectric properties, it can be seen that the value of the dielectric constant is linked to the orientation of the polarization, when the orientation of the polarization increases, the dielectric constant also increases, since the orientation of the polarization is due to the polarization. presence of permanent dipoles in each molecule [24]. It is necessary to understand that the presence of GTR (and more especially carbon black, the main component of the GTR) creates changes in the polymer structure. Thus, one of the important conclusions of this study is that carbon atoms are placed between the polymer chains, which indirectly causes a decrease in the chain-to-chain distance and, therefore, of the movement in the free volume. Thus, it can be deduced that changes in the structure created by the presence of the GTR in the polymer can also create changes (increases) in the moments of the dipole and, therefore, in the polarization and the measured dielectric constant. The study of the dielectric properties of polymers reinforced with GTR has shown that the presence of tires out of use in thermoplastic polymers increases the values of the conductivity and the tangent of delta. In

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each case, the evolution is due to changes in polarization (in particular to the orientation of polarization), through the observed changes in dielectric properties. The presence of the tire out of use causes the polarization to change by changing the structure and dipole moments of the polymer, especially thanks to the carbon black content.

5. CONCLUSIONS

First, the Tension-Deformation tests show how, by adding the GTR in the matrix, up to 10% percentages, the Young's modulus of the compound increases, although other mechanical properties decrease with the EVA, PVC and HDPE polymers. This behavior may be due to the fact that the reinforcement matrix is correct for these formulations and, therefore, some mechanical properties such as stiffness improve. However, for GTR concentrations greater than 10%, all mechanical properties decrease. The results obtained from the analysis of these compounds, determine as the GTR limit concentration 10-20%. From this concentration, the values of the mechanical properties begin to decrease, making it abruptly for high concentrations of GTR. This is, therefore, the mechanical limit that would allow the application of these compounds in various fields of industry, taking advantage of this material and saving new materials in various industrial applications in which the mechanical requirements are not excessive.

As for the dielectric parameters, it can be seen that they are significantly influenced by the GTR content from a concentration of 20% [25]. This behavior is attributable to the possible interaction between GTR particles when the concentration is greater than 20%. Basically, it is observed that the dielectric properties of the compound are maintained in values similar to those of the pure matrix of the polymers, up to approximately 10-20% GTR concentration. Since the interaction of the matrix with the GTR particle is quite low, this matrix accepts this low GTR content inside. For values greater than 20% in GTR, the dielectric properties decrease drastically.

Comparatively, it can be deduced from the results of HDPE, PVC, EVA, PP, PA6, ABS and PS with GTR, that the analyzed properties of the matrix undergo significant changes depending on the amount of GTR in the polymer matrix [26-27] (for a particle size of $p < 200\mu\text{m}$), concluding that the mixing limit value is of the order of 10-20% GTR. Mechanically, it is observed that HDPE, ABS, PS and PVC behave in a similar way, and instead, EVA and PP have a more flexible behavior than the rest of the polymers analyzed and, therefore, have a greater elongation at the break.

Once the results obtained from the mechanical and dielectric tests have been compared, with the standardized standard values appropriate for each industrial use, and except for the applications as dielectrics in special motors [28-32], it is concluded that the final application that will allow its use in The industry, in accordance with the regulations of the UNE Standards, is in our case the compound EVA+10%GTR which, according to the values obtained, could be used for the insulation of industrial footwear. Specifically, its parameters are within the limits established by these Standards: UNE-EN ISO 20345/6/7: 2005; UNE 53510: Conductivity $> 10^{-6}$ S/cm, Tensile strength $> 10-12$ MPa, and Elongation at break $> 450\%$.

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