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# Rheometric, transient, and cyclic tests to assess the viscoelastic behavior of natural rubber-based compounds used for rubber bearings

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ABSTRACT

The damping capacity of rubber bearings used as passive energy dissipators in bridges, buildings, and power generation plants is defined by the characteristic viscoelastic behavior of elastomers. Therefore, in this study, the viscoelastic behavior of natural rubber-based compounds used in rubber bearings with different content of carbon black is evaluated. Rheometric tests were performed to study the damping properties and curing parameters of the compounds. Transient tests, which are representative of the operating conditions of rubber bearings, were carried out to study the isochronous stress-strain relation evidencing a non-linear behavior under specific deformation states. Compression, shear, and tensile tests exhibited time-dependent mechanical properties. Moreover, energy dissipation in compression and shear increased for the higher carbon black content compound. Compression set exhibited permanent deformation of the compounds tested at accelerated temperature conditions. Experimental results obtained in this work have great importance for designers in order to evaluate the damping and mechanical behavior of natural rubber compounds related to rubber bearings performance.

## 1. Introduction

Laminated rubber bearings act as passive energy dissipators in structures and are widely used due to their high vertical stiffness, lateral flexibility, simplicity, and low maintenance requirements compared to alternative isolation systems [1], [2]. They serve as connectors between girder and pier in structures such as bridges and viaducts supporting large static loads due to the weight of the structure and dynamic loads due to vehicular traffic, wind, and thermal expansion, and since the 70 s are considered as seismic isolation systems [3], [4]. The damping capacity of rubber bearings depends on the viscoelastic behavior of elastomeric materials, operation parameters, and environmental factors. The dual response of an elastic material that obeys Hooke's law and a viscous fluid that obeys Newton's law is the cause of the viscoelastic behavior; hereby, the stress-strain relation is sensitive to time. Furthermore, this relationship tends to become non-linear at high deformations; nevertheless, there is no agreement to define the limit between high and low deformations [5].

Experimental results are essential to assess the damping properties to either characterize the material or for model validation purposes [6]. In experimental phases, standardized tests like dynamic-mechanical analysis (DMA) or transient tests such as stress relaxation and creep, are used in damping models. Those models include linear viscoelastic models, complex modulus approach, deformation energy-based models, and physics-based models [7]. Damping properties are related to the dissipated energy due to processes such as molecular mobility, break and reformation of the filler transient network, and slippage of rubber molecules under high strain amplitudes; however, part of the dissipated energy is transformed into heat, deteriorating the material properties [8]. DMA is used to study damping properties of elastomers obtaining tan $\delta$, which is defined as the ratio of the loss modulus ($S''$) to the storage modulus ($S'$). The storage modulus, or the in-phase component, represents the instant response to a stress or strain and it is related to the elastic behavior of the material, while the loss modulus, or the out-of-phase component, represents the dissipated energy as heat and it is related to the viscous response [8]. Natural rubber (NR) [9], recycled rubber [8,10], nitrile butadiene rubber (NBR) [11], silicone rubber-ethylene propylene diene monomer rubber blends (SR/EPDM) [12], and materials such as foams, cork, and rubbers for sound and vibration control [13] have been studied using DMA. According to de Silva [1] damping factors between 0.10 and 0.20 (considered as high-damping rubber bearings) are usually observed in seismic isolation bearings. However, moving die rheometry (MDR) is commonly used as a rheometric technique to evaluate the vulcanization process and, at an

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industrial level, have lower cost and are easy to analyze not only vulcanization parameters but also the damping factor. And even obtaining tan $\delta$ from MDR at a determined temperature and a specific shear rate, results can be useful in relation to viscoelastic properties. Using MDR, studies of the vulcanization parameters such as the scorch time ($t_{s2}$), the time at which 90 % of the vulcanization process has occurred ($t_{90}$), and maximum and minimum torques ($M_H$ and $M_L$ respectively) have been carried out [14–18].

Experimentally, when describing the viscoelastic behavior is required, it is preferable applying step stresses or strains (creep and stress relaxation respectively) instead of a ramp, in which constant stress or strain rates are applied because the time effect is isolated from any nonlinearity [19]. Time-dependent stiffness and energy dissipation due to cyclic loading are other phenomena inherent to the viscoelastic behavior of elastomers [19]. Understanding these rubber properties play an important role between structural engineers and rubber technologists in the selection of the rubber compound for elastomeric bearings [9]. Some studies have provided relevant information to understand the viscoelastic behavior of rubbers under different loading conditions. Oman and Nagode [20] performed uniaxial creep and stress relaxation tests to natural rubber-polybutadiene-styrene butadiene rubber (NR-BR-SBR) blends assuming that both processes are the result of the same viscoelastic mechanism. Yamaguchi et al. [21] studied the relation among creep, stress relaxation, and recovery of NR without fillers since they provide a higher complexity in the behavior of elastomers.

In this study, the viscoelastic behavior of natural rubber-based compounds reinforced with carbon black and typically used for rubber bearings is assessed through moving die rheometry, compression set, and mechanical properties evaluated through cyclic uniaxial compressive and shear tests, tensile tests, and transient tests such as stress relaxation and creep performed at room temperature. Experimental results obtained from laboratory tests to standardized specimens are evaluated and can be related to the damping behavior and time-dependent mechanical properties of rubber compounds used for laminated rubber bearings.

## 2. Experimental

### 2.1. Materials

Three natural rubber-based compounds typically used in elastomeric bearings and reinforced with 15, 30, and 45 phr of N550 carbon black were studied (hereafter called NR15, NR30, and NR45 respectively). The compounds were obtained in an open two-roll mill using the same ingredients with only the carbon black content varying among them. Table 1 shows the ingredients used in the formulation with a semi-efficient vulcanization system since conventional vulcanizing systems have a poor reversion resistance resulting in a larger drop in properties compared to efficient vulcanizing systems, in which the optimum state of curing takes a longer time. Compounds used for rubber bearings normally use a semi-efficient vulcanizing system, which possesses characteristics between both conventional and efficient systems [22]. 20 phr of Rubbersil RS-200® precipitated silica were added to the rubber compounds as a process aid, to improve the adhesion of rubber to metals, and to decrease heat build-up. Precipitated silica and N550 carbon black have been used in isolator compounds [9], and this silica content is usually used in combination with carbon black as reinforcement filler and, in this study, no coupling agent was used since the optimum silica loading in the absence of a coupling agent is close to 20 phr [23]. Although silica affects certain properties, changes in the behavior of the compounds evaluated in this study are attributed to changes in the carbon black content.

**Table 1**
Ingredients used in the formulation, phr (parts per hundred of rubber by mass).

| Ingredient | phr |
|---|---|
| Natural rubber, SGR10 (standardized Guatemalan rubber) | 100 |
| Antiozonant, 6PPD (N-(1,3-Dimethylbutyl)-N'-phenyl-p-phenylenediamine) | 1.5 |
| Antioxidant, TMQ (2,2,4-Trimethy1-1,2-Dihydroquinoline) | 1 |
| Paraffinic oil | 15 |
| Silica, RS-200® | 20 |
| Zinc oxide | 5 |
| Stearic acid | 1 |
| Wintag95® | 10 |
| Sulphur | 2 |
| Accelerator, CBS (n-cyclohexyl -2- benzothiazole sulfonamide) | 1.5 |
| Accelerator, TMTD (Tetramethyl thiuram disulfide) | 1 |
| Santogard®, PVI (N-(Cyclohexylthio) phthalamide) | 1 |
| Carbon black, N550 | 15, 30, 45 |

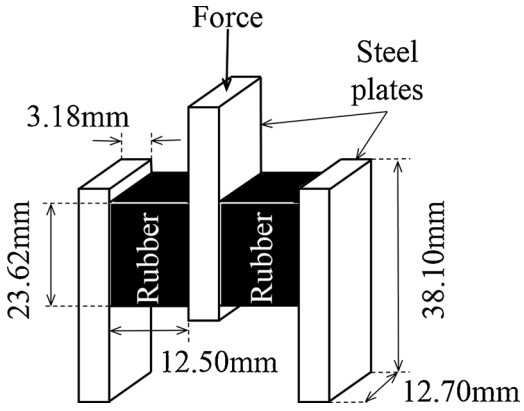


**Fig. 1.** Scheme of the shear specimens.

Cylindrical specimens of 29 ± 0.5 mm in diameter and 12.5 ± 0.5 mm in height of the compounds were obtained according to the recommendations of ASTM D575 [24] standard. Cylinders were vulcanized using a hydraulic press at 160 °C at an approximate pressure of 4.6 MPa. The vulcanization time was 33 min based on a $t_{100}$ of 9 min. Pure shear specimens (Fig. 1) were manufactured according to ASTM D945 [25] standard using the three compounds. Finally, vulcanized sheets of 150 × 150 mm and 2 mm thick were obtained with a hydraulic press at 160 °C for 9 min based on the same $t_{100}$ at an approximate pressure of 0.9 MPa. From the vulcanized sheets, dumbbell specimens were cut for uniaxial tensile tests according to ASTM D412 [26] standard.

### 2.2. Testing methods

Five samples of the uncured compounds were analyzed in a Pioneer Alpha Technologies MDR at 160 °C for 25 min, an oscillation amplitude of 0.5°, and a frequency of 1.66 Hz according to the recommendations of ASTM D5289 [27]. The samples were randomly selected taking into account the inherent variability of natural rubber as a plant origin material in addition to the variability associated with the mixing process.

Cylindrical specimens were used for uniaxial cyclic compression, transient, and compression set tests. Deformation and force-controlled uniaxial compressive tests were performed at rates of 1, 12, and 25 mm/min and 10, 50, and 100 N/s respectively. The third consecutive loading cycle was used to calculate the compression modulus and energy dissipation since the first two cycles serve as conditioning of the specimen. Sandpaper between the specimen surfaces and steel plates of the testing machine was used to prevent slippage [24]. Additionally, stress relaxation and creep tests in compression were performed to cylindrical specimens for 10 h following the recommendations of ASTM D6147 [28] applying different stress and strain levels. Compression set tests were performed using a BINDER air circulating oven according to

ASTM D395 standard (method B) [29], in which cylindrical specimens were compressed approximately 25 % during 70 h at 30, 50, and 70 °C.

Pure shear specimens (Fig. 1) were manufactured according to ASTM D945 [25] standard. The specimens were tested applying a vertical force to the middle steel plate while maintaining fixed lateral plates, at deformation and force-controlled rates of 1, 12, and 25 mm/min and 10, 25, and 50 N/s respectively. Three consecutive loading cycles were applied and the third cycle was used to calculate the shear modulus and energy dissipation.

Tensile tests were performed at deformation rates of 10, 200, and 500 mm/min until rupture. Tensile strength, modulus at 100 and 300 % of tensile strain (M100 and M300, respectively), and elongation at break were calculated.

All compressive, shear, and tensile tests were performed at room temperature using a SHIMADZU AGS series universal testing machine with a 50 kN load cell and a resolution of 0.01 N.

Shore A hardness was measured in the vulcanized cylindrical specimens using a CEAST durometer following the recommendations of ASTM D2240 [30].

## 3. Results and discussion

### 3.1. MDR

A typical rheometric curve with reversion obtained from MDR is shown in Fig. 2. The *S'* and *S''* curves associated with the storage and loss modulus respectively, and several curing parameters such as the scorch time $t_{s2}$ or the time when the crosslinks start to form, $t_{90}$ time in which 90 % of the vulcanization has progress, $M_L$ minimum torque, and $M_H$ maximum torque are illustrated in Fig. 2. The initial low values of *S'* curve in Fig. 2 are related to the uncured state of the compounds with an amorphous structure ($M_L$ values corresponding to the minimum value of *S'*) and at the end of the vulcanization reaction or crosslinking process the structure reaches the highest crosslink density ($M_H$ values corresponding to the maximum value of *S'*).

Fig. 3 shows the curves of *S'* and *S''* obtained during the vulcanization process from MDR of the compounds. As expected, the required torques *S'* and *S''* during the crosslinking process, associated to both the elastic and viscous components respectively, are greater for the NR45 compound due to the stiffening effect of the carbon black content, since the reinforcement particles restrict movements of the elastomer chains in both the elastic and viscous response of the specimen subjected to shear. For all the compounds, the vulcanization reaction starts between 0 and 2 min, in which the processability of the raw compound is related to the viscosity and heat transfer to the specimen. During the vulcanization process (crosslinking), where the elastic component *S'* increases, the viscosity of the compound is reduced because sulfur reactions take place and a crosslinked structure is formed. Furthermore, increased values of *S'* are observed as the carbon black content increase from 15 to 45 phr since the amount of immobilized rubber trapped in the filler network increases, which behaves as part of undeformable fillers, resulting in an increase of *S'* (known as the hydrodynamic effect) [8]. After $M_H$ is reached, reversion is observed, which has an undesirable effect on product quality [31]. Reversion of the rheometric torque curve has a significant effect on the final mechanical properties of the vulcanizates since degradation processes occur. The reversion rate was calculated as the slope of the linear regression in the reversion zone of the rheometric curve of each specimen. Mean values of the reversion rate are 0.0946 ± 0.0041, 0.1598 ± 0.0140, and 0.1223 ± 0.0106 dN m/min for the NR15, NR30, and NR45 compounds respectively, exhibiting significant differences at the 95 % confidence level. These values exhibit a slightly faster reversion rate of the elastic component (*i.e.*, reduction in the mechanical properties) of the compounds with higher carbon black content once the maximum of the vulcanization reaction is reached. This increased reversion rate of reinforced natural rubber is because carbon black increases the heat transfer rate [32].

As the vulcanization reaction initiates, the damping factor tan$\delta$ decreases (Fig. 4) between 0 and approximately 4 min indicating that before the crosslinked structure is formed during the vulcanization, there is a greater contribution of the viscous phase of the compounds; and as the reaction proceeds, the storage modulus, which is related to the elastic response of the material, increases; therefore, the damping factor decreases. Approximately in 9 min, when the vulcanization reaction is complete, the damping factor tan$\delta$ is 0.0629 ± 0.0010, 0.0818 ± 0.0143, and 0.1102 ± 0.0037 for the NR15, NR30, and NR45 compounds, respectively (see also Table 2) exhibiting significant differences. This effect is because larger amounts of carbon black in the compounds increase damping properties [9] because the filler transient network is the dominant factor governing the damping of the vulcanizates [8]. Using DMA, Koupai et al. [9] analyzed different rubber blends, which included natural, polybutadiene, and styrene-butadiene rubbers used for seismic isolator compounds with Shore A hardness between 48 and 67; and the obtained values of tan $\delta$ values were between 0.15 and 0.35 measured between 2 and 8 % of shear strain and a frequency of 0.2 Hz. In this work, Shore A hardness of the vulcanized compounds were: 47.2 ± 0.8, 56.2 ± 0.8, and 63.6 ± 0.5 for the NR15, NR30, and NR45 compounds respectively.

Using the smaller scale in Fig. 4, it is evident that the damping factor increases slightly in the reversion zone of the rheometric curves

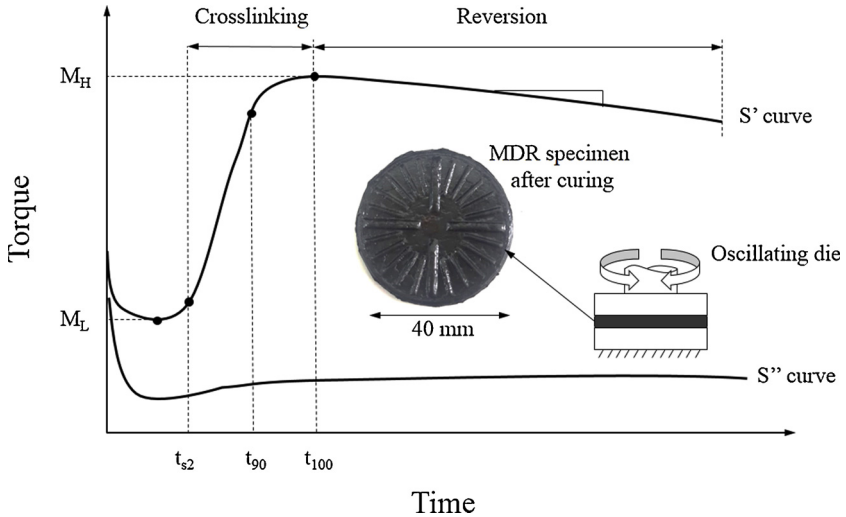


**Fig. 2.** Typical curves of *S'* and *S''* obtained from MDR.

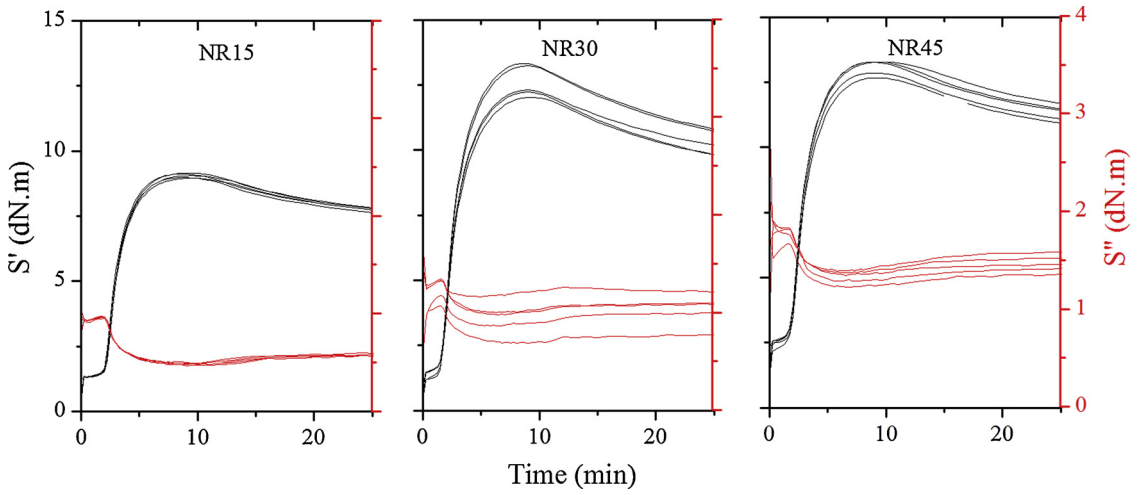


**Fig. 3.** MDR results at 160 °C results of the five replicates of each compound.

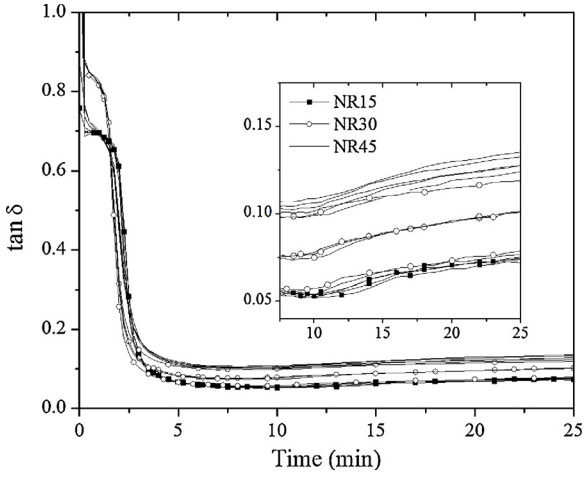


**Fig. 4.** tan $\delta$ curves of the compounds.

for all compounds because degradation yields in decreases of *S'* due to the destruction of polysulfidic crosslinks followed by the scission of main chains [31] but also due to slight increases of *S''* as shown in Fig. 3.

Table 2 shows the results of $t_{s2}$, $t_{90}$, $M_L$, and $M_H$. It is shown that $t_{s2}$ is slightly higher for the NR15 compound, and 90 % of the vulcanization reaction is reached faster than the other compounds; these results are associated with the accelerating effect of the reinforcement content on the vulcanization kinetics [33]. Curing parameters obtained in this study are comparable to those obtained in [16] where the vulcanization characteristics of natural rubber compounds with greater amounts of carbon black at different vulcanization temperatures were evaluated. Moreover, loss and storage modulus are also comparable to those obtained for nitrile and natural rubber blends [14].

### 3.2. Stress relaxation and creep

Fig. 5 shows the stress relaxation curves under a step uniaxial compression fixed to 15, 30, and 45 % of strain during 10 h using the procedure of standard ASTM D 575 [24] and applied by da Rocha et al. [34]. The stress normalized to the maximum initial stress is presented for all compounds. Since the stress $\sigma(t)$ is a function of time and strain level is held constant $\varepsilon_0$, the relaxation modulus $E(t) = \sigma(t)/\varepsilon_0$ decreases with time. On the other hand, creep results under uniaxial compression stresses fixed to 0.4, 0.8, and 1.2 MPa are shown in Fig. 6. Similarly, strain normalized to the maximum initial strain is presented. The creep compliance defined as $D(t) = \varepsilon(t)/\sigma_0$ increases with time under a constant stress $\sigma_0$ [35]. The difference in the strain values for each stress level between both compounds is due to the stiffening effect and greater resistance to creep as the carbon black content and stress level increase [36].

The isochronous variation of the stress-strain relation in transient tests allows defining linearity in the constitutive response of the material [35]. Hence, Fig. 7a shows the isochronous stress-strain diagram at $t_1$ = 1 h, $t_2$ = 5 h, and $t_3$ = 10 h from the stress relaxation data indicating a non-linear stress-strain behavior independent of the filler content of the compounds. Similarly, in Fig. 7b, the isochronous stress-strain diagrams from the creep tests are plotted using the same times ($t_1$, $t_2$, and $t_3$) evidencing a more linear behavior of the compounds; therefore, the creep compliance $D(t)$ is independent of the stress $\sigma_0$ [35]. It is observed in Fig. 7 that the response of the compounds to a specific deformation is highly non-linear, while at a specific load the response can be assumed as more linear. Since typical deformations of rubber bearings used in bridges (and it is known by experimental observations to a viaduct supported of bridge bearings by the authors) do not exceed 15 %, creep is expected to occur due to the weight of the structure and a linear stress-strain behavior over time for the stress levels used in this work can be assumed.

**Table 2**
Vulcanization parameters of the compounds from MDR.

| | $t_{s2}$ (min) | $t_{90}$ (min) | $M_L$ (dN.m) | $M_H$ (dN.m) | tan $\delta$ @ $t_{100}$ |
|---|---|---|---|---|---|
| NR15 | 2.54 ± 0.05 | 5.15 ± 0.09 | 1.32 ± 0.01 | 9.06 ± 0.09 | 0.0629 ± 0.0010 |
| NR30 | 1.91 ± 0.01 | 5.34 ± 0.12 | 1.31 ± 0.13 | 12.62 ± 0.61 | 0.0818 ± 0.0143 |
| NR45 | 2.15 ± 0.03 | 5.25 ± 0.07 | 2.41 ± 0.15 | 13.07 ± 0.29 | 0.1102 ± 0.0037 |

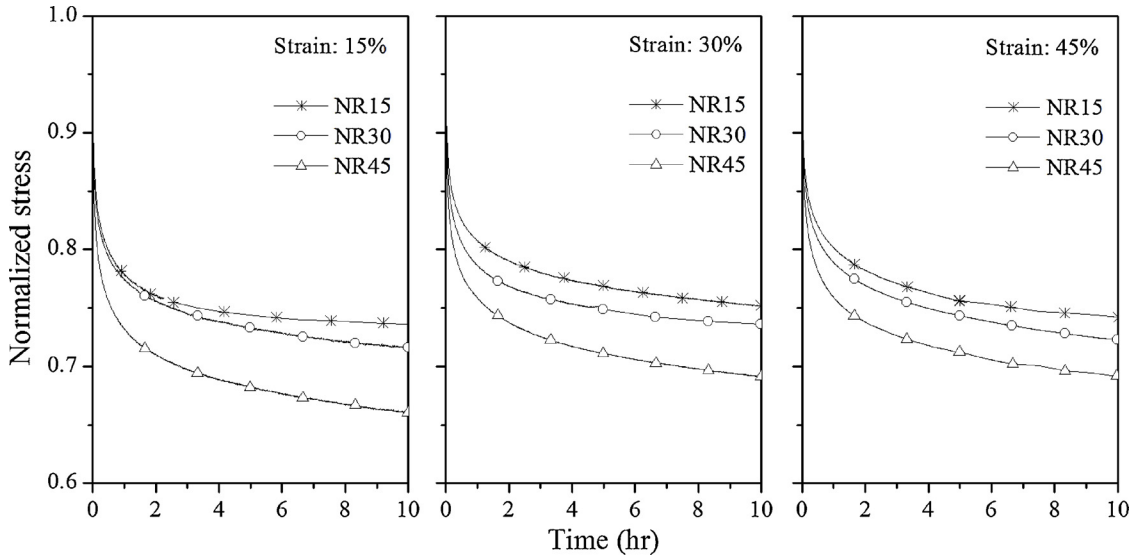


**Fig. 5.** Stress relaxation results at compressive strains of 15, 30, and 45 %.

### 3.3. Compression and shear tests

Tables 3 and 4 show a summary of the compression and shear modulus of both compounds at different deformation and force rates. Both the compression and shear moduli (initial tangent modulus obtained from the stress-strain curves depicted in Fig. 8) were calculated from the third loading cycle since stress softening or Mullins' effect [37] occur, in which a lower stress is required after applying the first loading cycle to reach the same strain level. However, the stress asymptotically approaches a steady state with an equilibrium of the stress-strain curve. This effect is more evident as the content of carbon black as reinforcement increases [37,38].

It is observed from the compression and shear tests results (Tables 3 and 4, and Fig. 8) the stiffening effect that the carbon black content has on the compounds, exhibiting significant differences (at the 95 % confidence level) in the compression and shear moduli among the compounds. As the reinforcement content increases, the mechanical properties measured in compression and shear increase. In particular, the compression modulus is a function of both material properties and geometry of the specimen [39]. In applications such as rubber bearings, which are mainly subjected to compression, the geometry of an individual rubber layer (*i.e.* the shape factor) is an important factor that influences the vertical stiffness and is a key parameter in their design [40]. For relative thin, cylindrical rubber layers, where the radius to height ratio is greater than about 5, the effective compressive modulus is much larger than the real value and is comparable in magnitude to the bulk compression modulus, and compressibility needs to be considered [39,41]. Furthermore, cylindrical specimens used in this study have a radius to height ratio close to unity to determine accurately their time-dependent compressive properties.

Constraining slippage of the loaded surfaces of the specimen yields in barreling or bulging, a phenomenon typically observed in laminated rubber bearings due to the restriction of slippage of the inner rubber layers. And part of the dissipation of the input energy can be expended in frictional sliding of the contact surfaces [42] instead of the inherent damping capacity of rubber components. Moreover, an additional stiffness due to the kinematic constraint is developed; thereby, a higher magnitude of the load is required to produce the same strain level compared to unbonded surfaces [43–46]. Since current standards regulate only uniaxial compression and simple shear tests due to the lack of availability of procedures for biaxial compression [47], uniaxial compressive tests carried out in this study were performed following the

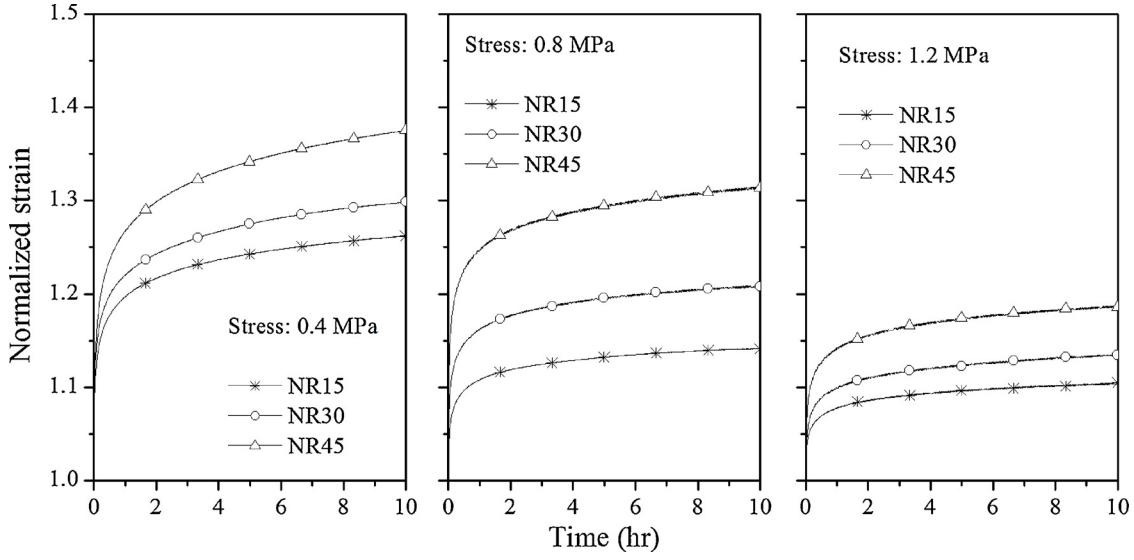


**Fig. 6.** Creep results at compressive stresses of 0.4, 0.8, and 1.2 MPa.

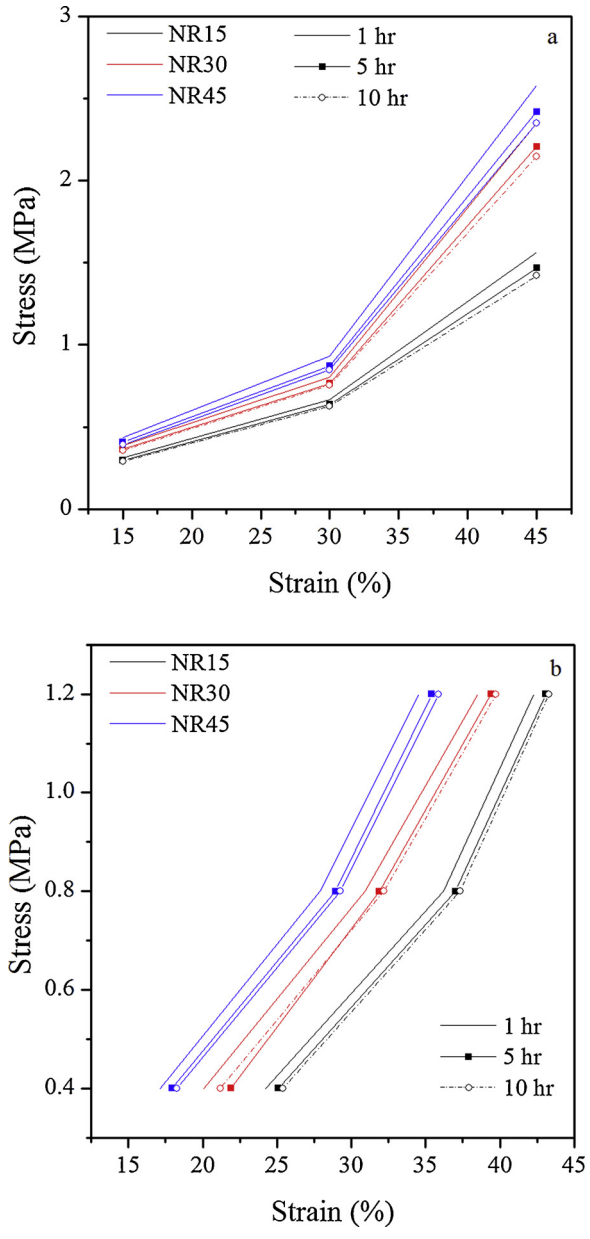


**Fig. 7.** Isochronous stress-strain diagrams for $t_1$ = 1 h, $t_2$ = 5 h, and $t_3$ = 10 h from the (a) stress relaxation and (b) creep tests.

recommendations of ASTM D575 [24] also used by [34], [48], and for modeling purposes [49,50]. Furthermore, during homogeneous uniaxial compression, the principal stretch in the loading direction becomes compression, and in the other two directions become tension; and, considering isotropy and incompressibility, the principal stretches will depend only on the compressive stretch [51]. Thereby, using the methodology of ASTM D575 standard, the dependence of the mechanical properties on the deformation and force rates due to viscoelastic effects of the compounds can be observed especially in the compression modulus (Table 3), in which significant differences are exhibited (at the 95 % confidence level) between 1 and 25 mm/min, and 10 and 100 N/s. To a lesser extent, in shear, the correspondent modulus of NR15 shows significant differences between 1 and 25 mm/min, and 10 and 50 N/s (Table 4). As the deformation or force rate increases, the material tends to stiffen since the elastomer molecules do not have enough time to take a new configuration and there is no reformation of the filler transient network, processes that are associated with energy dissipation [8]. Additionally, at higher strain rates, strain-induced crystallization can occur if strain crystallization time scales are comparable [52].

From Fig. 8, a marked non-linear behavior is evidenced [44] (especially in compression) of the NR45 compound, which can be attributed to material and geometric non-linearities, which include bulging. On the other hand, the NR15 compound, which has the lower carbon black content, a more linear behavior is observed in the loading cycles.

### 3.4. Energy dissipation

Using the third cycle, the energy dissipation, which is related to the damping capacity, is calculated and corresponds to the area enclosed by the hysteresis loop [53]. The energy dissipated by the compounds in the cyclic compressive and shear tests is shown in Tables 5 and 6 respectively. As expected, energy dissipation increases as the carbon black content increases. From both results of energy dissipation in compression and shear, and the damping factor tan $\delta$ from MDR, it is observed that increasing the carbon black content, both the energy dissipation and damping properties increase (exhibiting significant differences at a confidence level of 95 % among the compounds) due to mobility and slippage of rubber molecules, and break and reformation of the filler transient network [8]. Also, in the rheometric curves (Fig. 3), when the vulcanization reaction was completed (approximately 9 min), the elastic component $S'$ is predominant allowing the component with higher carbon black content to absorb more energy during cyclic shear in MDR. This effect is confirmed by the energy dissipated during cyclic shear presented in Table 6, in which significant differences are evidenced among the rubber compounds. And, although tan $\delta$ from MDR is measured at 160 °C and a frequency of 1.66 Hz, while cyclic tests are carried out at room temperature and at a much lower frequency, results exhibit a relation between tan $\delta$ and the energy dissipated during cyclic tests. In shear, energy dissipation exhibits significant differences within each compound only for the force-controlled tests. Significant differences in the mean values of the dissipated energy in compression are observed between 1 and 25 mm/min, and 10 and 100 N/s within each rubber compound.

### 3.5. Compression set

Table 7 shows the compression set or permanent deformation remaining after a compressive deflection of 25 % of the cylindrical standardized specimens. Compression set is expressed as a percentage of the original deflection and intends to measure the ability of rubber components to retain their elastic properties after a long-term compression under accelerated temperature conditions [29]. Values of 0 %

**Table 3**
Summary of compression modulus (in MPa) of the compounds.

| | Deformation rate (mm/min) | | | Force rate (N/s) | | |
|---|---|---|---|---|---|---|
| | 1 | 12 | 25 | 10 | 50 | 100 |
| NR15 | 2.24 ± 0.06 | 2.43 ± 0.08 | 2.49 ± 0.02 | 2.37 ± 0.06 | 2.56 ± 0.08 | 2.75 ± 0.13 |
| NR30 | 3.41 ± 0.20 | 3.55 ± 0.05 | 3.50 ± 0.15 | 3.19 ± 0.20 | 3.44 ± 0.13 | 3.83 ± 0.05 |
| NR45 | 4.33 ± 0.20 | 4.61 ± 0.15 | 4.81 ± 0.13 | 3.91 ± 0.09 | 4.33 ± 0.14 | 4.52 ± 0.09 |

**Table 4**
Summary of shear modulus (in MPa) of the compounds.

| | Deformation rate (mm/min) | | | Force rate (N/s) | | |
|---|---|---|---|---|---|---|
| | 1 | 12 | 25 | 10 | 25 | 50 |
| NR15 | 0.88 ± 0.03 | 0.99 ± 0.08 | 0.98 ± 0.07 | 0.67 ± 0.03 | 0.78 ± 0.05 | 0.87 ± 0.06 |
| NR30 | 1.41 ± 0.11 | 1.47 ± 0.06 | 1.65 ± 0.14 | 1.31 ± 0.13 | 1.40 ± 0.04 | 1.45 ± 0.16 |
| NR45 | 2.20 ± 0.45 | 2.21 ± 0.43 | 2.25 ± 0.37 | 1.74 ± 0.15 | 1.83 ± 0.24 | 2.22 ± 0.41 |

mean a fully recovered height, while 100 % means no recovery [54]. Results show significant differences between mean values of compression set at all temperatures evaluated within each compound. However, only significant differences between the NR15 and NR45 compounds are observed at each temperature. Higher contents of carbon black yield in increased compression set as obtained by Koupai et al. [9]. Compression set values obtained at 70 °C are comparable to those obtained in [15] for compression set carried out at 100 °C for modified NR compounds. These results exhibit that during the design process of the rubber compound in bearings applications, as the reinforcement content increases due to energy dissipation requirements, an increased permanent deformation is expected.

### 3.6. Tensile tests

Tensile properties shown in Table 8 are comparable to those obtained for natural rubber blends [14,15,17]. Additionally, as the carbon black content as reinforcement filler increases, tensile properties, specifically the M100 and M300 moduli, increase, as obtained by Fan et al. [16] since fillers impart high stiffness or hardness to rubber products [15]. It is exhibited that as the deformation rate increases, both the M100 and M300 moduli tend to increase especially for the low carbon black content compound, in which significant differences were observed. Furthermore, despite tensile strength showed no significant differences among the compounds, elongation at break was significantly reduced as the carbon black content of the compounds increase from 15 to 45 phr.

## 4. Conclusions

The viscoelastic behavior of natural rubber-based compounds typically used for rubber bearings was evaluated through rheometric, transient, and cyclic tests. Furthermore, testing standardized specimens at different deformation and force rates allowed identifying the influence of time on the mechanical properties of the compounds. The following conclusions can be drawn:

- MDR allowed identifying that the damping factor among the

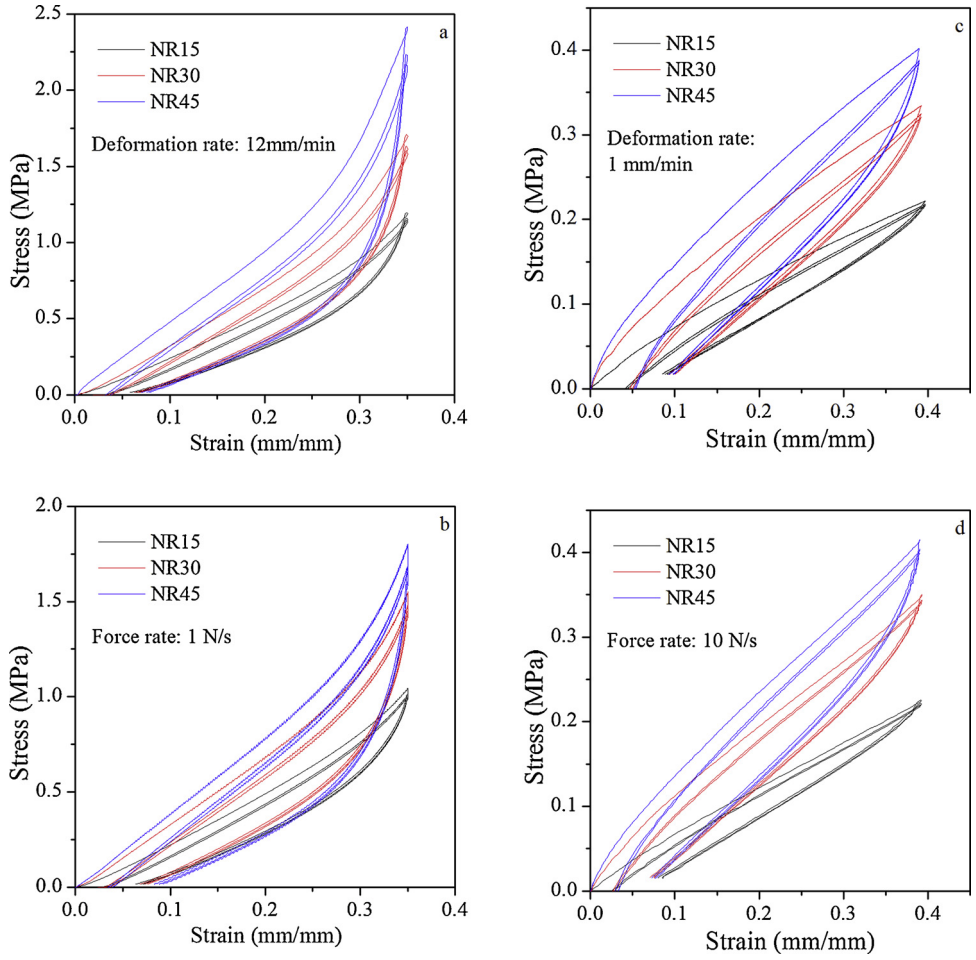


**Fig. 8.** Stress-strain curves of the compressive (a and b) and shear tests (c and d).

**Table 5**
Energy dissipation (mJ/mm$^3$) in compression.

| | Deformation rate (mm/min) | | | Force rate (N/s) | | |
|---|---|---|---|---|---|---|
| | 1 | 12 | 25 | 10 | 50 | 100 |
| NR15 | 3.4E-2 ± 1.9E-3 | 4.2E-2 ± 2.2E-3 | 4.1E-2 ± 3.8E-3 | 4.0E-2 ± 1.9E-3 | 4.8E-2 ± 1.8E-3 | 4.8E-2 ± 2.6E-3 |
| NR30 | 5.7E-2 ± 2.9E-3 | 6.9E-2 ± 4.9E-3 | 7.3E-2 ± 5.7E-3 | 6.4E-2 ± 3.4E-3 | 7.3E-2 ± 3.6E-3 | 7.4E-2 ± 3.1E-3 |
| NR45 | 8.5E-2 ± 0.3E-3 | 11.0E-2 ± 1.5E-3 | 11.2E-2 ± 3.1E-3 | 9.7E-2 ± 1.2E-3 | 11.3E-2 ± 2.8E-3 | 11.7E-2 ± 1.4E-3 |

compounds studied has increased values for the compound with higher carbon black content. The technique provides, in addition to the parameters related to the vulcanization process, information related to the damping characteristics in bearings applications. Despite hardness is a commonly used technique in the rubber industry, the authors encourage the use of MDR as a viscoelastic characterization technique to determine damping properties (which are related to the dissipated energy) in addition to curing parameters. According to the results obtained in this study, natural rubber-based compounds with 15, 30, and 45 phr of carbon black content as reinforcement, which exhibited an overall shear modulus of 0.86 ± 0.12, 1.45 ± 0.15, and 2.07 ± 0.37 MPa (NR15 and NR30 compounds within the accepted range of AASHTO specifications [20]) showed damping factors at $t_{100}$ of 0.0629 ± 0.0010, 0.0818 ± 0.0143, and 0.1102 ± 0.0037 respectively. Moreover, Shore A hardness of the compounds was 47.2 ± 0.8, 56.2 ± 0.8, and 63.6 ± 0.5, of which the NR30 and NR45 compounds are accepted in the AASHTO specifications.

- Overall energy dissipation in compression of the compounds were 0.0424 ± 0.0057, 0.0683 ± 0.0073, and 0.1058 ± 0.0116 mJ/mm$^3$ as the carbon black content increased 15, 30, and 45 phr respectively; and in shear: 0.0071 ± 0.0018, 0.0150 ± 0.0018, and 0.0214 ± 0.0017 mJ/mm$^3$. However, significant differences were observed as the deformation or force rate varies and, in applications such as bearings, changes in external vibration frequencies may produce variations in the dissipated energy.
- Stress relaxation and creep tests under uniaxial compression were carried out to standardized specimens allowing to assess the time dependence of the relaxation modulus and creep compliance due to the viscoelastic behavior of the compounds. A non-linear behavior to specific strains (stress relaxation) and a more linear behavior under specific loads (creep) was observed. Since rubber bearings are typically subjected to creep, a linear stress-strain behavior may be assumed independent of the filler content of the compounds for the stress levels used in this work.
- Mechanical behavior in compression, shear, and tension was studied at different ramp applied deformations and stresses, and besides the time-dependent mechanical properties observed, a non-linear behavior under compression was observed unlike the isochronous stress-strain diagrams for creep tests that indicate a more linear behavior. Nevertheless, rubber bearings are subjected to both overlapped loading states, the former due to cyclic loading such as traffic and the latter due to the weight of the structure. And their viscoelastic response will be determined by the frequency of the cyclic loading generated by vibration due to traffic, wind or seismic activity.
- Compression set values showed significant differences among the compounds. As the carbon black content increased, the compounds exhibited differences in long-term-accelerating compressive conditions due to differences in the carbon black content. Therefore, required damping properties and long-term compression should not only meet hardness design standards requirements but can be useful and should be included as design criteria of rubber bearings.
- Increased sensitivity of the time-dependent properties is more evident in the compressive and shear tests, and in the NR15 compound compared with tensile results. However, tensile tests can be used to check the properties from one batch to another. Therefore, as a design procedure, compressive and shear tests are more appropriate in selecting the proper rubber compound for laminated bearings.

**Table 7**
Compression set (%) of the compounds at different temperatures.

| | Temperature | | |
|---|---|---|---|
| | 30 °C | 50 °C | 70 °C |
| NR15 | 16.45 ± 0.46 | 23.92 ± 1.14 | 41.99 ± 1.31 |
| NR30 | 16.42 ± 0.89 | 22.80 ± 0.65 | 42.64 ± 0.39 |
| NR45 | 21.55 ± 1.81 | 28.65 ± 3.65 | 44.49 ± 1.43 |

In this work, the experimental results obtained are of great importance to study the damping properties of rubber compounds related to the viscoelastic behavior of rubber components used as isolation systems not only for applications in rubber bearings but also in machinery and equipment for the control of vibration. Also, the results can be used to validate phenomenological constitutive models for validation purposes. Finally, structure movements and vibrations, in addition to temperature conditions, may also increase the internal temperature in service. More studies are required in this regard.

**Table 6**
Energy dissipation (mJ/mm$^3$) in shear (x10$^{-3}$).

| | Deformation rate (mm/min) | | | Force rate (N/s) | | |
|---|---|---|---|---|---|---|
| | 1 | 12 | 25 | 10 | 25 | 50 |
| NR15 | 6.8E-3 ± 6.6E-4 | 7.9E-3 ± 7.2E-4 | 8.3E-3 ± 7.1E-4 | 3.7E-3 ± 8.4E-4 | 7.3E-3 ± 2.2E-4 | 8.8E-3 ± 9.3E-4 |
| NR30 | 14.3E-3 ± 9.2E-4 | 15.9E-3 ± 1.3E-3 | 15.8E-3 ± 0.8E-3 | 12.7E-3 ± 2.2E-3 | 14.2E-3 ± 0.4E-3 | 17.0E-3 ± 1.2E-3 |
| NR45 | 20.2E-3 ± 0.6E-3 | 21.9E-3 ± 1.2E-3 | 20.1E-3 ± 1.2E-3 | 19.7E-3 ± 1.1E-3 | 22.7E-3 ± 0.8E-3 | 23.5E-3 ± 0.6E-3 |

**Table 8**
Tensile properties of the compounds.

| | Deformation rate (mm/min) | Tensile strength (MPa) | M100 (MPa) | M300 (MPa) | Elongation at break (%) |
|---|---|---|---|---|---|
| NR15 | 10 | 13.75 ± 0.82 | 1.27 ± 0.03 | 4.40 ± 0.18 | 611.46 ± 10.48 |
| | 200 | 15.24 ± 0.50 | 1.40 ± 0.07 | 4.88 ± 0.29 | 627.00 ± 21.38 |
| | 500 | 15.34 ± 1.01 | 1.45 ± 0.05 | 5.04 ± 0.22 | 632.71 ± 32.11 |
| NR30 | 10 | 14.27 ± 0.21 | 2.04 ± 0.08 | 7.06 ± 0.34 | 503.13 ± 14.90 |
| | 200 | 13.81 ± 1.20 | 2.21 ± 0.03 | 7.65 ± 0.12 | 474.52 ± 36.57 |
| | 500 | 13.84 ± 1.40 | 2.28 ± 0.19 | 7.48 ± 0.54 | 492.92 ± 21.84 |
| NR45 | 10 | 13.52 ± 0.53 | 2.44 ± 0.13 | 8.04 ± 0.37 | 456.28 ± 12.49 |
| | 200 | 13.43 ± 0.72 | 2.43 ± 0.12 | 8.04 ± 0.37 | 463.81 ± 23.95 |
| | 500 | 13.53 ± 0.32 | 2.43 ± 0.01 | 7.94 ± 0.10 | 478.51 ± 8.69 |

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## CRediT authorship contribution statement

**Manuel Alberto Guzmán Sánchez:** Conceptualization, Methodology, Formal analysis, Investigation, Writing - original draft. **Diego Hernán Giraldo-Vásquez:** Methodology, Resources, Writing - review & editing, Validation. **Ricardo Moreno Sánchez:** Writing - review & editing, Supervision.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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