RESEARCH ARTICLE

INTERNATIONAL RESEARCH JOURNAL OF
MULTIDISCIPLINARY TECHNOVATION

Asian Research Association

DOI: 10.54392/irjmt26316

Design and Characterization of a Cyclic Stress Imposed Accelerated Aging Test Method – Assessment on Tyre Tread Compound

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DOI: https://doi.org/10.54392/irjmt26316

Received: 17-01-2026; Revised: 07-04-2026; Accepted: 25-04-2026; Published: 19-05-2026

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Abstract: Surface mobility has been one of the greatest enablers of human development, and tyres have played a pivotal role in this journey. The tyre is the only link between the vehicle and the road to enable safety. As a major requirement, the traction is achieved by the tyre with the help of several components of its structure. The tread patterns provide physical interlocking with road asperities and the tread compound interfaces with road textures, to effect necessary grip in dynamic conditions. As the properties of tread compound play the most vital role in affecting dynamic grip, the aging of the rubber and consequent loss of properties have become key topics to study in tyre design for safety and durability assessment. Tread ageing is affected by multiple simultaneous conditions experienced by the tyre, related to thermo-oxidative exposure and mechanical stress variations. While aging is a critical influencer in tread performance preservation, the studies related to aging have made many assumptions due to difficulties in replicating actual service conditions in the laboratory. Traditionally, aging studies were conducted by inducing a thermo-oxidative environment at an elevated temperature above ambient temperature and mapping property degradation as a function of time. This generic approach was fraught with many limitations due to its inability to replicate the degradation characteristics as they happen in real service conditions. A novel attempt is made to develop a facility that simulates real-life service conditions to a greater extent by imposing a cyclic mechanical stress during the accelerated aging process. This study investigates the effect of aging on Natural Rubber (NR) based tyre tread compound under conditions dominant in dynamic service conditions. Unlike conventional accelerated aging without mechanical stimuli (may be called static aging), the current study involves cyclical mechanical stress imposed on the tread compound under elevated temperatures (dynamic aging) to mimic the effect on tyre tread in service conditions. This paper addresses the synergistic interactions mechanical behaviour of tread rubber compound and its microstructure alterations when exposed to static and dynamic aging processes. Various methods in mechanical and viscoelastic studies to verify representative parameters of rubber compound behaviour, such as hysteresis ratio, Mullins coefficient, stress relaxation, and macromolecular network alteration, are introduced. A macromolecular network-alteration mechanism, linking changes in crosslink density and chain scission to the mode and state of aging, is established. The findings significantly reinforce the understanding of compound behaviour alterations with more realistic operating conditions of aging to support the design of materials to enhance product safety.

Keywords: Tyre, Braking force, Tread compound, Thermo-oxidative aging, Cyclic mechanical stress, Dynamic aging.

1. Introduction

Oxidative aging can induce significant changes in the dynamic mechanical properties of elastomers across operating conditions. These changes can weaken the critical requirements of the tread element to meet its designed performance and, in extreme situations, result in catastrophic failures. Elastomer components make up about 40-65% of the tyre weight, and the component that comes in direct contact with the road surface, called tread, and significantly influences the safe performance of the vehicles [1]. Design specifications of tread compound are arrived after meticulous studies and evaluations to deliver conflicting tyre performance requirements of rolling resistance, wet grip, and abrasion resistance (long-term durability). Notably, these properties are greatly linked to the composition of the tread compound, which is significantly altered under the influence of thermal and oxidative aging during actual service conditions. This is in line with many of the recent reviews, which highlighted how rubber formulations can play a critical role in optimizing the above-mentioned mutually conflicting performance elements. The durability of tread compounds over aging is influenced by the interactions between fillers and rubber, as well as the resultant network structure. Improved interfacial coupling and co-crosslinking in silica-filled systems can affect the compound's stability and its resistance to degradation under working conditions [2].

Rubber aging is a time-dependent phenomenon that often extends to a significant time scale, forcing the designers to delay the product design finalization and industrialization. The understanding of conditions required to accelerate the aging process thus assumes significance in the related research. However, necessary caution must be exercised to guarantee that the accelerated testing environments replicate conditions analogous to the field aging of components [3-5].

Rubber components are suitable for their intended use primarily due to their dynamic flexibility and elasticity. Rubber aging may expedite damage and reduce service life based on the operating conditions. Particularly for tyre tread rubber to provide traction, dynamic flexibility is one key requirement to envelop road asperities at different speeds and contact (grip) frequencies. Unfortunately, rubber components with progressive aging lose their flexibility and become rigid, rendering them ineffective in intended applications. Thermal aging is a significant factor that accelerates tread rubber deterioration to a loss of tire performance [6].

Rubber components utilize their viscoelastic properties to perform particularly performance levels. Viscoelasticity induces hysteresis losses, which raise temperatures within the structure. This, along with ambient thermal conditions, can accentuate thermal oxidation, which is a crucial factor for degradation. However, thermal oxidation is an unavoidable component of the product lifecycle, including in the stages of manufacturing, processing, storage, application, and disposal.

The primary challenge for any product made out of polymeric materials is to preserve its performance through a prolonged usage life in the presence of the synergistic but detrimental effects of heat and oxygen. Oxidative degradation in polymers initiates upon the interaction of oxygen with the polymer surface, resulting in the fragmentation of macromolecular chains. Thermo-oxidative aging is the primary cause of degradation in commercial tires. Oxygen diffusion into the material breaks the chemical bonds inside the rubber matrix [7]. The process of aging, which typically initiates with radical generation, then interacts with oxygen to accelerate the degradation pathway. Circumstances that can trigger oxidative degradation are many, namely Ultraviolet (UV) radiation or mechanical stress, apart from oxygen and high temperature exposures. More importantly, thermal oxidation (oxygen-induced breakdown at elevated temperatures) can extend into the material bulk, and it is more detrimental than photo oxidation, which confines itself to the superficial layer just under the incident surface layer [8-9]. Prior to interacting with the polymer structure, oxygen molecules are initially absorbed by the surface layer and subsequently distributed throughout the material. All polymeric materials contain a substantial amount of peroxide radicals created through polymerization and processing stages. The continuation of oxidation is subject to slower mechanisms, particularly the removal of hydrogen from polymer molecules in the vicinity by the peroxide radical [10]. But breakage of primary chain bonds of the macromolecule happens when thermal energy exceeds the bond dissociation energy. Also, the structural defects in the materials support the process by breaking the chains and producing free radicals that trigger chain propagation mechanisms. However, the speed and extent of diffusion are influenced by various factors, including temperature, molecular structure, and external conditions such as exposure time and oxygen supply. These interdependency of components poses great difficulty in the precise prediction of oxidation behaviour in polymer aging models [11].

Zhang *et al*. [12], studied the effect of thermal aging of vulcanized natural rubber (NR) that negatively impacts the ultimate tensile strength and elongation at break. However, the disparity in strength between aged and unaged rubbers under equivalent loading circumstances was small. In a distinct study, styrene-butadiene rubber (SBR) samples underwent oven aging at 80 °C for periods of 4 and 6 weeks [6]. The post-aging tensile strength of these samples exhibited a significant reduction relative to their initial values. It was found that maintaining a specific oxygen concentration threshold was vital for sustaining appropriate reaction kinetics. Tyre, essentially being a container of air, the availability of oxygen in the cavity decreases with extended temperature exposure in an oven [13]. Although it is widely recognized that tire rubber undergoes oxidation due to the pressurized air inside the tire as well as from oxidation resulting from ambient air around the tyre, the focus on the aging because of the latter mechanism has been much less. A study involving two radial tyres from a reputable brand, fitted on an Airbus aircraft, encountered multiple incidences of tread separation, which was triggered by oxidation from both inside and outside.

Thermal degradation of rubber from tires occurs in two phases. The initial phase involves crosslinking, while the subsequent phase promotes chain scission. The transition occurs more rapidly in the presence of oxygen, resulting in accelerated crack formation and deteriorated mechanical characteristics [14]. Xie *et al.* [15] assert that increased temperatures accelerate the development and spread of micro-cracks, undermining the mechanical integrity of rubber under stress, to finally result in failure. As a result, thermally aged rubber initiates a cascade of material deterioration, resulting in reduced stiffness and performance, which results in substantial compromise in the performance and longevity of the affected tyres. Thermo-oxidative aging and mechanical fatigue can significantly alter the mechanical properties of elastomer compounds in service. The interaction of these factors is complex, as it involves modifications in crosslink density, chain scission, polymer-filler interactions, and deterioration of the filler network [16].

However, the effect of thermal aging of tyres under dynamic loading is yet unclear, and much less exploration has been done for the complexity and investments required for necessary infrastructure development. A few studies were carried out to stimulate thermal ageing of the sample under the application of cyclic load using a universal testing machine UTM) to simulate dynamic ageing on the rubber compound. But such efforts still fail to represent the actual application of tires. To bridge this gap, our research work concentrates on the study of the effect of static aging (Heat) vs dynamic aging (Dynamic load and Heat using a facility developed as part of the study) to compare the performance properties of either specimen on LAT- 100 using annular-shaped rubber samples. Annular ring shape of the samples helps to run the sample on the rotating road wheel of the dynamic aging machine and subsequently on the test surface of the LAT 100. The adopted test method has a significant correlation to the actual application of tires.

2. Experimentation

The compound under investigation is a functional tire tread formulation for commercial vehicles, with its composition expressed as parts per hundred rubbers (phr). EQ Rubber supplied the base polymer, which was natural rubber (NR; 100 phr), specifically of the Technically Specified Rubber grade TSR-10. The primary reinforcing filler used was carbon black (CB) grade N134, obtained from Philips Carbon Black. It was designated as Super Abrasive Furnace (SAF; 50 phr) black, having a nitrogen surface area of 138.42 m^2/g. The secondary reinforcing filler is highly dispersible silica (HD silica; 5 phr) with a nitrogen surface area of 182.54 m^2/g. The formulation contained various ingredients, including paraffin-based wax (PE wax; 2 phr) for ozone protection, the antioxidant and antiozonant N-(1, 3-dimethylbutyl)-N-phenyl-p-phenylenediamine (6PPD; 2 phr), and the anti-degradant 2, 2, 4-trimethyl-1, 2-dihydroquinoline polymer (TMQ; 2 phr). Additional compunding ingredients comprised stearic acid (SA; 1 phr), zinc oxide (ZnO; 5 phr), curing agents (DPG; 1.5 phr), N-tertiary-butyl-2-benzothiazole sulfenamide (TBBS; 1.2 phr), and soluble sulfur (S; 1.5 phr) acting as the vulcanizing agent.

2.1 Preparation of rubber compounds

The initial stage was to formulate a masterbatch consisting of rubber and reinforcing fillers. A subsequent phase followed, during which curatives were incorporated to prepare the final batch. The masterbatch was blended utilizing a Farrell HF series Banbury mixer, which has a mixing chamber capacity of 4.79 liters. In this study, the parameters used are rotor speed (60 rpm), fill factor (70%), and initial chamber temperature (100 °C). The rubber is masticated for 1 min in mixer followed by the addition of other compound ingredients like filler, ZnO, stearic acid, antioxidants, anti-ozonates, etc., and mixed for 3 mins. The compound mixed for 6 minutes was collected and stored at room temperature for 24 hours. After the maturing period, the master batch and curatives were further mixed in Banbury with rotor speed (30 rpm), fill factor (70 %), with the temperature of mixer at 70 °C for a duration of 3 minutes. The final mixed batch is moulded as per standard procedure for slab and LAT100 samples [17].

2.2 Static and Dynamic Ageing

The molded LAT 100 samples were subjected to static and dynamic aging using the designed dynamic aging machine as shown in Figure 1, which consists of an oven chamber with rotating disc. The term Dynamic aging describes the samples that are exposed to Aging under continuous cyclic stress. Static aging represents that the samples are exposed to conventional accelerated aging without any cyclic stress (conventional method).

2.3 Dynamic Aging Machine - Design and Operation

The aging machine is designed to simulate the effects of long-term use and environmental exposure on rubber materials, while also applying mechanical stress. This allows for a more realistic and comprehensive assessment of the material's durability and performance.

The machine consists of two main components:

1 Environmental Chamber: This chamber controls the temperature, humidity, and other environmental conditions, such as exposure to UV light or ozone.

2 Mechanical Stress Unit: This unit applies a controlled mechanical stress to the rubber samples.

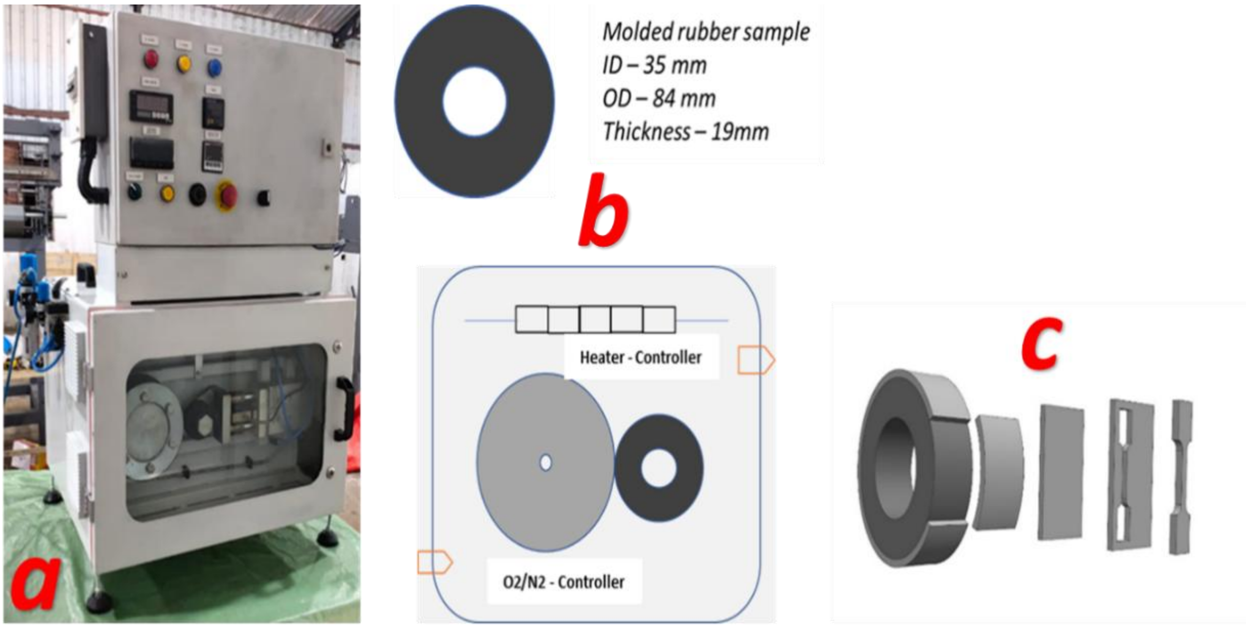

Figure 1 (a). Designed dynamic aging machine for LAT 100 samples. **(b)** Schematic representation of the samples for dynamic loading **(c).** Sample extraction for the testing.

The stress can be static (constant load) or dynamic (cyclic load), and can be applied in various modes, such as tension, compression, or shear. Constant load on the rotating sample becomes cyclic loading at the contact.

The rubber samples are placed in the environmental chamber and subjected to the desired environmental conditions. Simultaneously, the mechanical stress unit applies the programmed stress regime to the samples. The machine can be programmed to run for extended periods, allowing for accelerated aging studies.

Key Features and Specifications

- Temperature Range: 30 °C to 100 °C
- Force Range: 50 to 100 N
- Stress Modes: Continuous loading
- Stress Control: Rotational speed range 15 to 25 km/hour
- Sample Capacity: 2 samples dynamic and 2 samples static holding capacity

The whole setup has control over the temperature, humidity, and environmental control. Both the samples, yet to be exposed to static and dynamic aging conditions, were kept in the same chamber, whereas the samples for the dynamic aging were attached to the setup where the load was applied along with the rotation of the wheel at a specific speed. Both static and dynamic aging take place at a temperature of 70 °C for specific days. (Figure 1). The oven chamber is positioned and vented to the laboratory atmosphere. The oven operates with forced convection using an internal circulation fan to ensure continuous air movement. Temperature uniformity within the chamber was verified by the oven manufacturer's specification ±2 °C across the working volume. It is part of the machine design and development assessment. The air exchange rate of the oven is estimated to be in compliance with the standard oven used for rubber samples, approximately 10 – 15 chamber volume changes per hour.

3. Characterization

Mechanical characterization was conducted via tensile testing to evaluate the influence of filler presence in the thermo-oxidatively aged rubbers on their mechanical properties, by making dumbbell test samples with a thickness of 2 mm, in accordance with ASTM D412-C standard. The tensile testing machine utilized for this investigation is a Zwick Z010 series, which contains a 1 kN load cell. Tests are performed at a standardized crosshead speed of 500 ± 50 mm/min in a controlled environment, maintained at a temperature of 23 ± 2 °C and relative humidity of 50 ± 5%. Elasticity was assessed by dynamic testing methods utilizing cyclic and multi-step loading, utilizing a proprietary methodology based at reduced loading rates of 100 mm/min and 50 mm/min. This method facilitated incremental stress application and enabled accurate assessment of viscoelastic responses in both unaged and thermo-oxidatively aged rubber specimens under various loading conditions. The detailed procedure was expressed in our earlier research article [17].

A loading rate of 100 mm/min was employed to allow a more gradual application of stress, facilitating a more precise evaluation of the material's viscoelastic behavior.

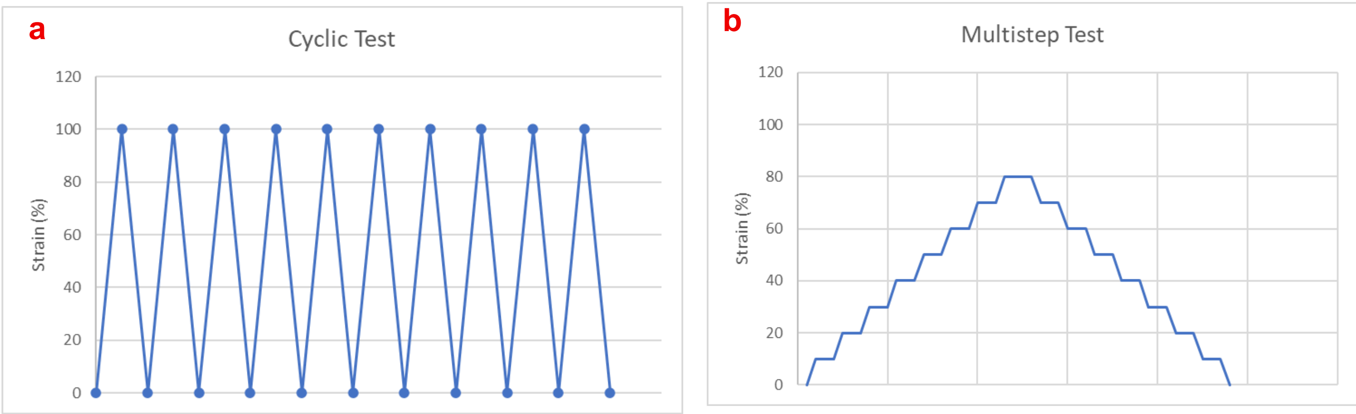

Figure 2. Schematic representation of the loading profile imposed on thermo-oxidatively aged and unaged rubber specimens. **(a)** Cyclic. and **(b)** Multi-step loading.

The unaged and thermo-oxidatively aged rubber specimens were subjected to the following loading profiles.

Cyclic Test: To investigate the evolution of Mullins softening, the aged and virgin specimens were subjected to a cyclic loading profile with 100 % constant maximum strain consisting of 10 cycles, as shown in Figure 2 (a). During the cyclic loading, a constant strain rate of 100 mm/min was maintained for each cycle.

Multi-step Test: To investigate the effect of viscoelastic response on thermo-oxidatively aged rubbers, the specimens (aged and unaged) were subjected to a multi-step loading profile, as shown in Figure 2 (b). At each step, a 10 % increment in load was imposed until a maximum strain of 80 % with a relaxation time of 180 seconds for both uploading and unloading paths. Prior to multi-step mechanical loading, the specimens were subjected to cyclic loading to remove the effect of Mullins softening. A constant strain rate of 50mm/min.

To further investigate the material's elastic and viscoelastic responses, dynamic testing, including cyclic and multi-step loading, was explored using an indigenous test method. A lower loading rate of 100 mm/min was employed to allow for a more gradual application of stress, which facilitates a more precise evaluation of the material's viscoelastic behavior. The unaged and thermo-oxidatively aged rubber specimens were subjected to the following loading profiles.

A DMA +1000 (METRAVIB, France) was used for dynamic mechanical analysis to analyze the viscoelastic properties, dependent upon strain and temperature. Temperature sweep tests were performed in tensile mode from −80 °C to 25 °C at 1% dynamic strain and 10 Hz frequency to estimate the glass transition temperature (Tg) and to measure the storage modulus, loss modulus, and the damping factor (tan δ) as functions of temperature. Strain sweep measurements carried out at 60 °C and 1 Hz, with strain amplitudes from 0.1% to 15%, quantified the Payne effect, indicative of filler network degradation under increasing deformation, thereby facilitating the assessment of nonlinear viscoelastic behavior and filler-polymer interactions in the cured rubber samples. Equilibrium swelling tests were conducted to compute the crosslink density. Initially, the dry, unaged, and aged samples were weighed m_i was recorded. Subsequently, the samples and the corresponding mass were immersed in Toluene for 72 hours m_s was measured. Finally, the samples and the dried mass were dried in an oven at 40 °C in a vacuum for 12 hours m_d was recorded. The following Flory-Rehner equation was used to determine the crosslink density (ν_c) of unaged and aged rubbers:

$$\nu_c = -\frac{\ln(1-V_r)+V_r+\chi V_r^2}{V_o\left(V_r^{\frac{1}{3}}-\frac{2V_r}{f}\right)} \quad (1)$$

where V_o is the molar volume of the solvent (for toluene =106.9 cm3/mol), V_r is the volume fraction of rubber in swollen rubber, f is the crosslinking functionality of the polymer (set to 4 with the assumption of tetra-functional crosslinks), and χ is the Flory-Huggins interaction parameter. The value of χ for the SBR-Toluene system was found to be 0.378 [18]. The filler correction factor is incorporated using the Kraus equation during the volume fraction measurement in swollen rubbers [19, 20].

Abrasion loss and braking force were evaluated using the LAT 100 system under the test conditions specified in Table 1. The Laboratory Abrasion Tester (LAT 100) is widely used to assess the wear resistance of rubber compounds by simulating tire–road interaction under controlled laboratory conditions. Disc-shaped specimens with an inner diameter of 35 mm, an outer diameter of 84 mm, and a thickness of 19 mm were prepared for the tests. During testing, a mixture of magnesium oxide powder and alumina (120 grit) was applied between the rotating track and the specimen surface to prevent smearing of the rubber surface. The experiments were conducted in accordance with ISO

23233 standards. All compound formulations were examined for key wear-related performance parameters, particularly braking force and abrasion loss, under varying speeds and slip angles.

4. Results and Discussion

Table 2 presents the physical properties of extracted samples (from LAT100 specimen) before as well as after aging for 2, 5, and 10 days under both static and dynamic conditions. The Shore A hardness increased with age, indicating that the material became stiffer. Dynamic aging resulted in a marginally lower hardness than static aging, presumably due to ongoing mechanical stress that causes chain breakage and hinders certain crosslinking processes [21]. The modulus values (M100, M200, M300) increased with aging. Static aging demonstrated a slight increase in stiffness [22, 23], whereas dynamic aging resulted in a more significant rise, particularly over longer exposure, suggesting that mechanical stress expedites network stiffening by facilitating crosslink formation and limiting chain mobility. A distinct divergence in the rate of hardness and modulus increase was also observed. Elongation at break and Tensile strength decreased with aging, with dynamic aging causing a greater decline than static aging. This reduction is attributed to matrix degradation from oxidation and chain scission, intensified under dynamic conditions where cyclic stress accelerates chain breakage and network weakening [24]. In summary, both static and dynamic aging increase stiffness and reduce flexibility, with dynamic aging exerting a stronger influence on mechanical degradation. Continuous mechanical stress accelerates chain scission, leading to greater brittleness, reduced tensile strength, and lower elasticity. These findings highlight the need to account for dynamic aging when assessing the long-term durability of rubber vulcanizates used in applications involving continuous mechanical loading.

The evolution of network changes is studied by the solvent swelling measurement. Figure 3 shows the crosslink density values measured for both static and dynamically aged samples. In both static and dynamic aging conditions, crosslink density increases gradually with an increase in the exposure duration. This trend is consistent with the typical thermo-oxidative behavior of natural rubber, where the oxidative coupling process generates additional crosslinks through radical recombination in the network. The cross-over is observed in the longer aging duration (10 days); the crosslink density in the dynamically aged sample shows only a marginal increase, remaining slightly lower than the value measured for the statically aged specimen. This behavior suggests that cyclic stress-imposed aging introduces an additional degradation pathway that complements oxidative crosslink formation. Mechanical loading can promote bond rupture and network rearrangement within the elastomer, leading to localized chain reorientation and chain scission. As a result, the net evolution of crosslink density under dynamic conditions reflects the combined influence of oxidative crosslink formation and mechanically assisted chain scission. To further understand the network changes, additional cyclic and viscoelastic measurements were done on the samples and discussed in detail.

Table 1. Testing conditions for the abrasion mass loss and braking force using LAT 100

LAT 100 Testing Condition				
Parameters	Normal load	Speed	Slip angle	Distance
Units	*N*	*km/h*	°	*m*
Abrasion Test	75	25	4 to 16	250 to 900
Braking Force Test	75	1	0	-

Table 2. Effect of static and dynamic aging on the tyre tread compound on Physical and mechanical properties using UTM

Properties	***Units***	***Unaged***	***S-2D***	***S-5D***	***S-10D***	***D-2D***	***D-5D***	***D-10D***
Hardness	***Shore A***	64-66	65-66	67-68	69-71	66-68	66-68	67-69
M100	***MPa***	2.2±0.05	2.5±0.06	2.6±0.06	2.84±0.05	2.63±0.04	2.69±0.06	3.07±0.08
M200	***MPa***	4.5±0.12	5.3±0.15	5.4±0.15	5.8±0.16	5.3±0.12	5.39±0.15	6.0±0.16
M300	***MPa***	7.74±0.15	8.8±0.20	8.9±0.30	9.5±0.21	8.1±0.23	8.7±0.35	9.5±0.45
Tensile Strength	***MPa***	26.6±0.75	21.2±0.9	22.7±0.5	21.9±0.8	23.5±0.8	20.9±1.2	18.9±1.2
Elongation at Break	***%***	771±9	634±10	637±15	631±10	634±9	606±15	534±12
R. I	**-**	3.52	3.52	3.42	3.35	3.11	3.26	3.12

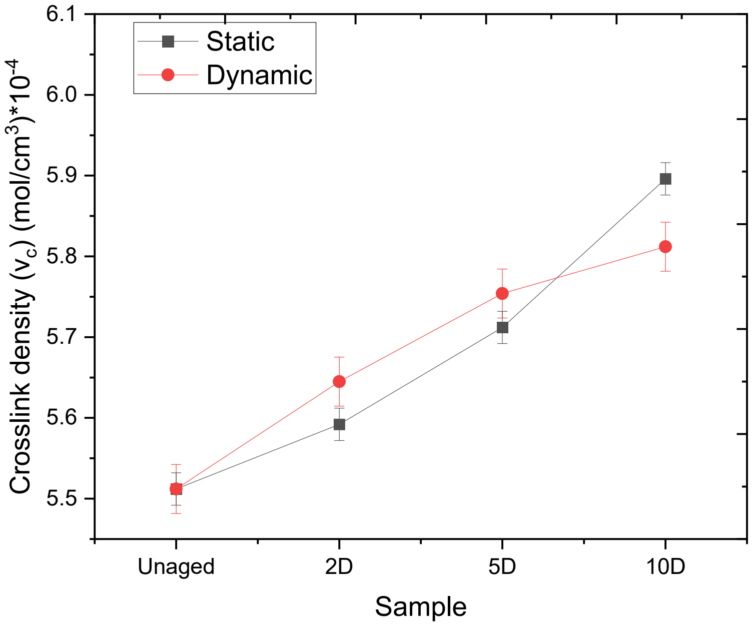

Figure 3. Crosslink density measured by the solvent swelling method for unaged and aged samples

4.1 Impact of Tensile Cyclic Response Analysis under Thermos-Oxidative Aging

To examine the evolution of the molecular architecture and network characteristics, the cyclic deformation, the engineered response for stress-strain behaviors, for unaged and aged samples (Static and Dynamic condition) were conducted. The uniaxial tensile direction at 10 cycles and 100 % strain was executed and represented in Figure 4a.

Invariably, the unaged and aged rubbers exhibit inelasticity, resulting in stress-softening, hysteresis, and permanent set. The effect of stiffness is found on the thermo-oxidatively aged rubbers, which may change the microstructure of the rubber and polymer-filled matrix. The inelasticity in the rubber is estimated by the Mullins effect, which is a function of strain energy density using the equation (2):

Stress-softening $\phi = 1 - \frac{A_2}{A_1}$ (2)

Where A_1 and A_2 are the areas under the curves of 1st and 2nd uploading cycles respectively. For a detailed description of the computation of stress-softening using equation (2). Figure 4b. shows the stress-softening effect estimated with strain energy density, which is a function of different aging conditions and duration. It is interesting to notice that statically aged samples (2 days and 5 days) show reverse stress softening. A similar observation was reported in our earlier work [17]. This behavior arises from the ability of polymer cross-linking with post-curing characteristics to reconfigure and strengthen during deformation. Conversely, dynamically aged samples show consistent and linear stress softening phenomenon as a function of exposure time. During tyre operation, repeated deformation of the rubber in the tread and sidewall regions occurs, which evidently causes a local energy distribution in the tyre, influencing traction, rolling resistance, and durability performance [25, 26]. We can observe a divergence in the behaviour of the statically and dynamically aged samples with special reference to the stress-softening behaviour. Static aging primarily induces chemical hardening through crosslinking, reducing the magnitude of the Mullins effect and limiting hysteretic energy losses [27]. Dynamic aging, on the other hand, imposes mechanical fatigue that enhances micromechanical damage and internal friction, leading to higher hysteresis and greater total energy dissipation.

4.2 Study of Thermos-Oxidative Aging for Relaxation Response in Multi-Steps

To evaluate the viscoelastic response, specimens underwent multi-stage relaxation loading according to the test profile. Before this sequence, they were cycled 10 times in uniaxial tension to eliminate the Mullins effect [28]. The resulting multi-step relaxation curves (Figure 5) show a pronounced stress relaxation effect in both rubber conditions. At each level, the value of stress has fallen during loading and rises during unloading, with better relaxation occurring on the loading path—particularly at higher strains. Figure 5.c presents the progression of equilibrium hysteresis for unaged and aged samples.

To study the hysteresis in thermo-oxidatively aged rubbers, the hysteresis loss ratio proposed by Bergstrom and Boyce [10] as referred to.

Hysteresis loss ratio $= \frac{H}{E}$ (3)

where E and H represent the area of the uploading cycle in the stress-strain diagram and the area under the stress-strain curve for the uploading and unloading path, respectively.

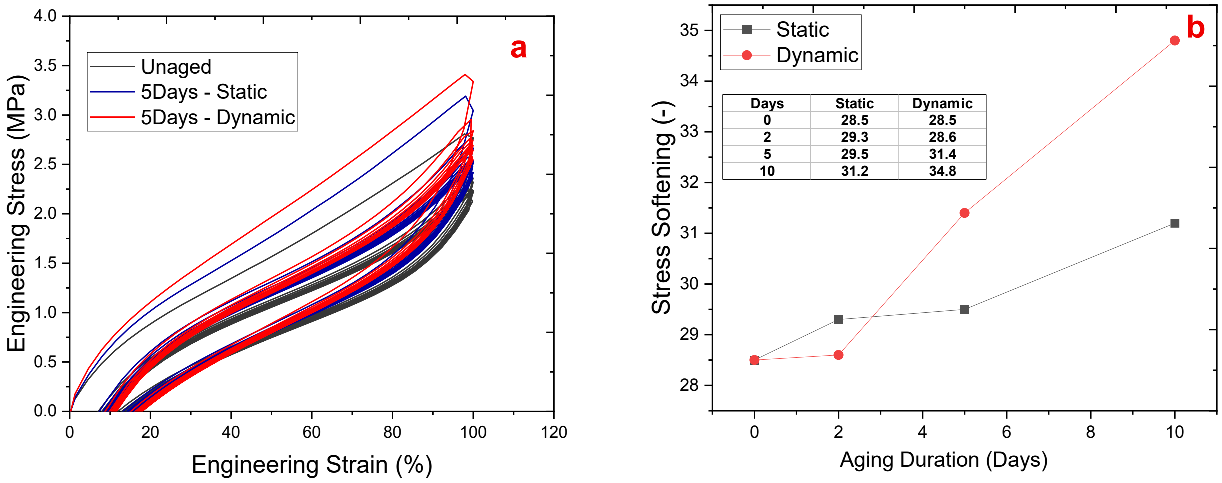

Figure 4 (a). Stress vs. strain graph for aged and unaged engineered rubbers, under tensile axial loading at 100 % strain in unaged, 10 days statically aged (10S), and 10 days dynamically aged (10D). **(b)** Stress-softening effect in aged and unaged rubbers at different modes of exposure.

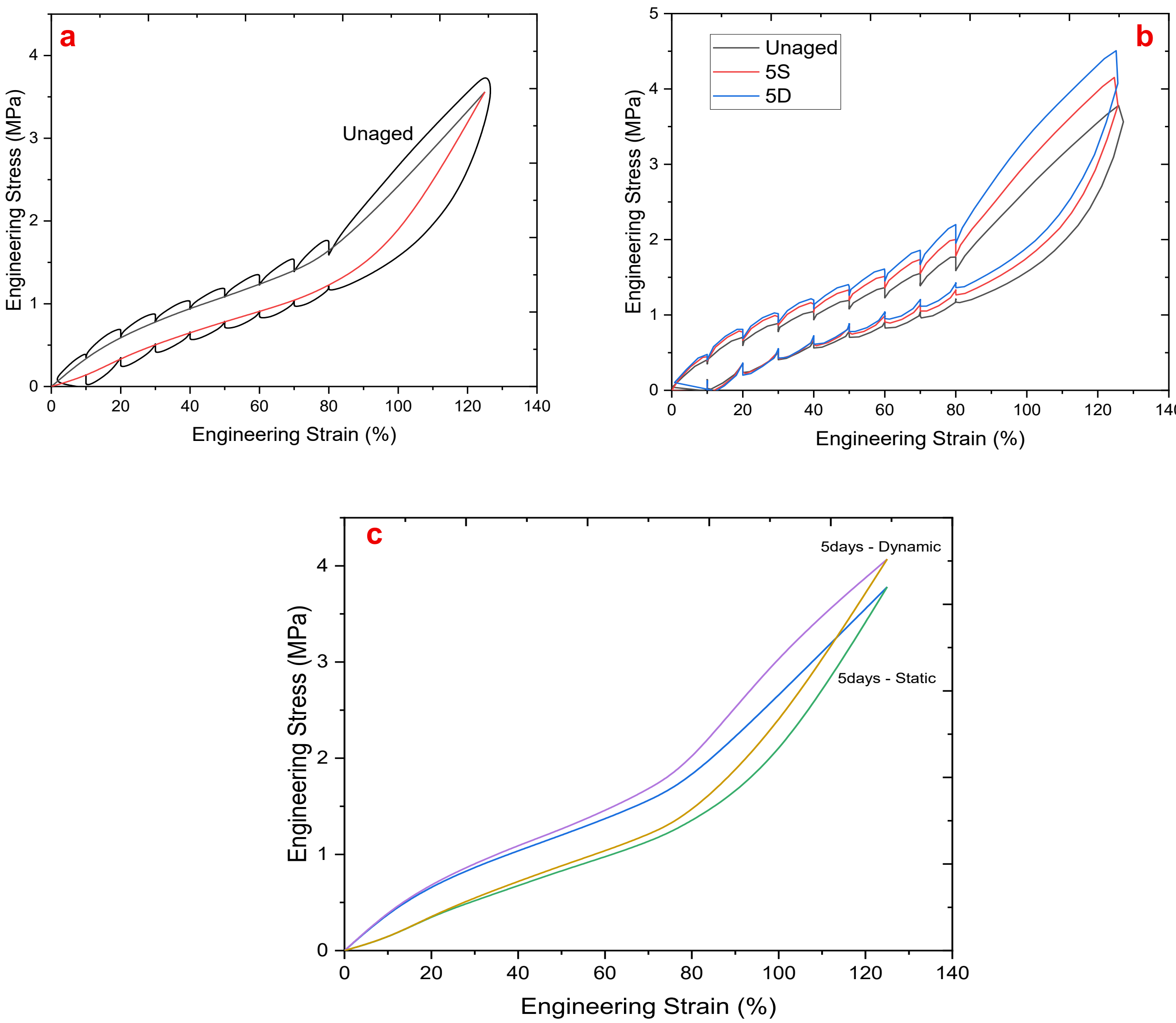

Figure 5 (a). Result of the multi-stage relaxation response impacting the tensile properties of rubbers. **(b)** Hysteresis loop of equilibrium condition for unaged rubbers **(c)** Comparison of equilibrium hysteresis for static and dynamic aged sample (5 Days).

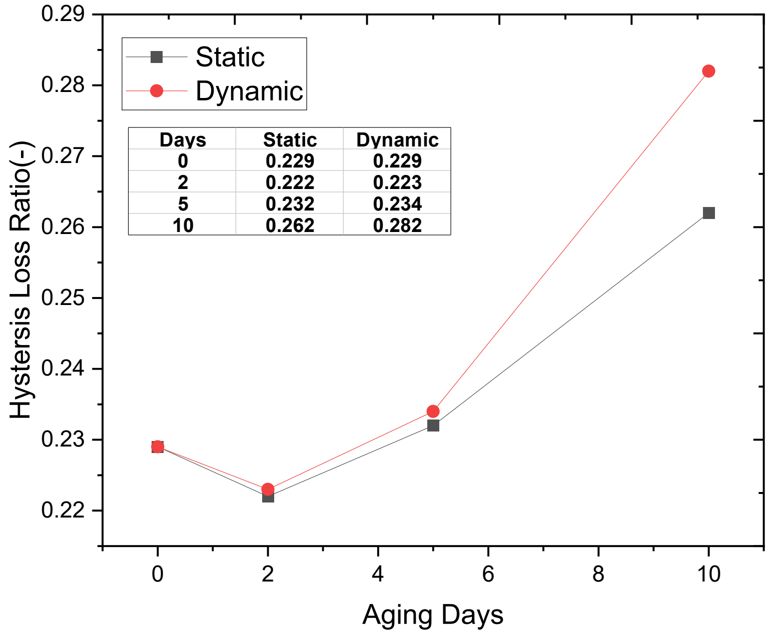

Figure 6. Evolution of hysteresis loss ratio for unaged and aged with different modes of exposure

Basically, hysteresis in unaged rubbers arises from the capacity of polymer chains and the filler network to reorient during deformation [29]. Figure 6 provides the experimental results of hysteresis in both rubbers with a change in the mode of exposure. It is found that hysteresis is affected by the entanglements or physical cross-links present in the elastomer network. The statically aged samples show a decrease in hysteresis, which can decrease the occurrence of entanglements because of predominant crosslinks [30]. The dynamically aged samples show an increase in hysteresis that might be due to an increase in entanglements, which is caused by chain rupture and micromechanical damage.

4.3 Dynamic Mechanical Properties

Inferring viscoelastic properties over a wide range of temperatures will provide insight into the molecular architecture due to aging (Figure 7). Static aging leads to an initial increase in storage modulus, particularly at high temperatures (+60 °C), suggesting post-curing effects or additional crosslink formation, which enhances rigidity. Under dynamic aging, storage modulus increases at early stages (2 days) but decreases over extended aging periods (10 days), both at the low temperature region (-30 °C) and at higher temperatures. This implies mechanical degradation due to chain scission and thereby reduces network integrity. The contrasting trends between static and dynamic aging highlight that thermal aging stiffens the material, whereas cyclic loading induces structural reorganization and density of the elastomeric network [31]. The glass transition temperature (Tg) of the unaged sample is recorded at -57.5 °C, indicative of the pristine segmental mobility. Static aging results in a slight increase in Tg with an increase in aging time, signifying network densification due to oxidation-induced crosslinking. Dynamic aging exhibits a more significant shift in Tg to higher temperatures (Table 3). Continuous cyclic mechanical loading accelerates structural changes mainly due to the competing reactions of chain scission, which reduces molecular weight, simultaneous oxidative reactions, and network rearrangement can increase the effective crosslink density and restrict chain mobility. Although chain scission occurs during thermo-oxidative degradation of natural rubber, oxidation also induces secondary reactions such as branching, and cyclization of polyisoprene chains. These processes restrict segmental mobility and increase the effective network stiffness, which results in a shift of the tan δ peak (associated with Tg) toward higher temperatures. Consequently, dynamically aged samples need higher onset thermal input to start the segmental mobility.

4.4 Laboratory Abrasion Tester (LAT100)

All experiments were conducted using a LAT-100 Laboratory Abrasion Tester equipped with a standardized corundum abrasive track (P60 grade). Prior to each measurement sequence, the track surface was cleaned with compressed air and conditioned with dummy clean-in samples to ensure the removal of dirt. The test temperature was maintained at 23 ± 2 °C under controlled laboratory conditions. The specimen temperature was monitored to avoid excessive frictional heating, and measurements were conducted under steady-state conditions.

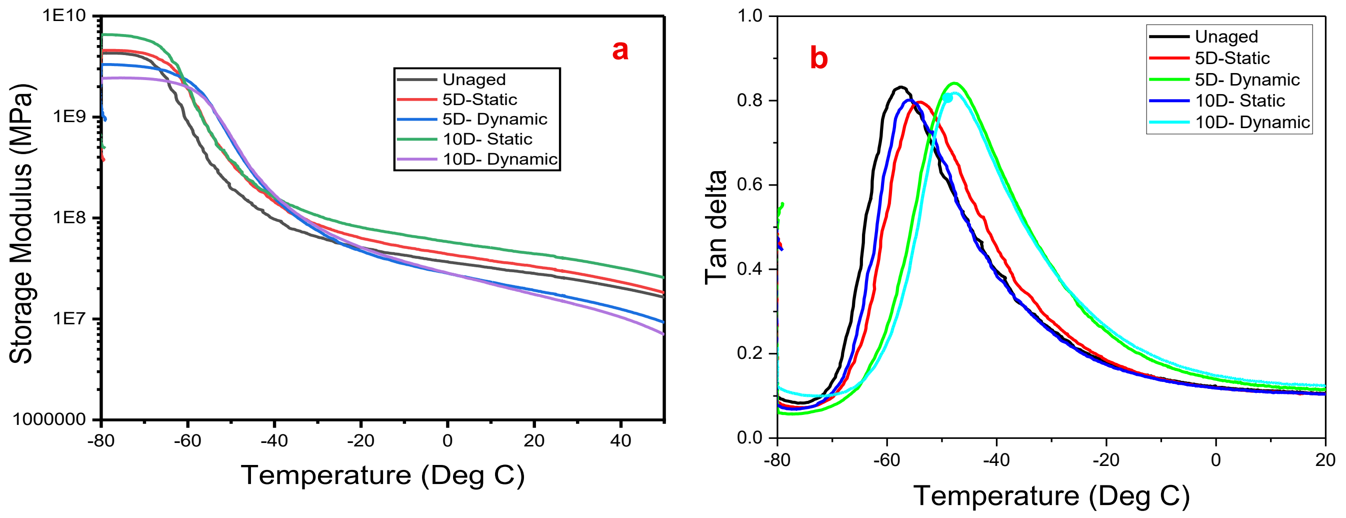

Figure 7. Temperature sweep of static and dynamic aged samples using DMA (a) Storage modulus vs Temperature, and (b) loss tangent vs temperature

Table 3. Storage modulus and Tan delta values for the samples at different temperature

Sample	-60 °C (MPa)	-30 °C (MPa)	0 °C (MPa)	30 °C (MPa)	Tan δ Peak	Tg (°C)
Unaged	910	65.2	37.3	24.3	0.8320	-57.5
S-2D	1790	97.5	46.6	28.1	0.7792	-54.5
D-2D	2865	90.1	39.2	23.2	0.8682	-51.3
S-5D	1980	86.4	44.4	28.6	0.7961	-53.9
D-5D	2325	75.5	28.3	16.4	0.8412	-47.8
S-10D	2056	110.1	58.4	38.7	0.8010	-56.1
D-10D	2102	81.3	28.5	14.4	0.8180	-47.6

A standardized powder application protocol was applied before each run, consisting of a thin and uniformly distributed silica powder layer to minimize rubber build-up on the abrasive surface and to ensure repeatable sliding conditions. For abrasion measurements, specimens were tested under constant normal load and slip conditions, and wear was quantified through mass loss after a defined sliding distance. Braking force and side-force measurements were obtained by imposing controlled longitudinal and lateral slip angles, respectively, while continuously recording the frictional force. To minimize systematic bias, all measurements were randomized across the different aging conditions. Samples from each aging state were tested in alternating order to avoid potential drift associated with track wear, environmental variations, or machine stabilization effects. In addition, replicate measurements were performed to confirm the reproducibility of the obtained results.

Abrasion resistance of natural rubber (NR)–carbon black tire-tread compounds is a critical performance metric, especially for truck and bus radial tires operating under severe service conditions. The LAT-100, a widely used controlled abrasion and friction tester, enables the prediction of compound response to varied load and speed conditions during tread development. Table 4 and Figure 8 shows LAT-100 abrasion volume-loss data for unaged (UA), statically aged (S-2D, S-5D, S-10D), and dynamically aged (D-2D, D-5D, D-10D) samples tested at four slip angles. The results confirm that accelerated aging measurably affects abrasion performance. Both thermal-oxidative (static) and stress-enhanced thermo-oxidative (dynamic) aging causes microstructural stiffening and embrittlement, increasing material removal. Dynamic aging shows a consistent trend: dynamically aged samples exhibit systematically higher abrasion volumes than unaged and statically aged counterparts at equal exposure times across all slip angles. In contrast, statically aged samples show irregular behavior with multiple value crossovers and no progressive trend. At this stage, the authors are not clear why static aging is not showing a trend; further analysis might help us to understand the behaviour.

Table 4. Summarized the LAT100 test results - Side force measurement and mass loss for the samples

Sample	Slip Angle (deg)	Distance (m)	Side Force (N)	Mass loss (mg)	Abradability (g/m)
UA	5.5	900	48	4.56	50.67
S-2D	5.5	900	47	4.32	48.00
S-5D	5.5	900	49	4.89	54.33
S-10D	5.5	900	50	4.83	53.67
D-2D	5.5	900	47	5.45	60.56
D-5D	5.5	900	44	5.75	63.89
D-10D	5.5	900	44	6.02	66.89
UA	9	750	55	8.45	112.67
S-2D	9	750	57	8.16	108.80
S-5D	9	750	58	8.12	108.27
S-10D	9	750	59	8.85	118.00
D-2D	9	750	57	10.14	135.20
D-5D	9	750	56	10.36	138.13
D-10D	9	750	53	10.96	146.13
UA	11	500	64	9.67	193.40
S-2D	11	500	66	9.23	184.60
S-5D	11	500	67	9.88	197.60
S-10D	11	500	70	10.16	203.20
D-2D	11	500	65	10.21	204.20
D-5D	11	500	63	11.23	224.60
D-10D	11	500	61	13.24	264.80
UA	16	350	70	11.12	317.71
S-2D	16	350	71	11.23	320.86
S-5D	16	350	70	11.56	330.29
S-10D	16	350	72	12.01	343.14
D-2D	16	350	62	12.34	352.57
D-5D	16	350	67	13.48	385.14
D-10D	16	350	64	14.65	418.57

A possible reason would be that the LAT100 sample is thick, and the static aging is competing with several reactions (scission, crosslinks, cyclization, oxidative-induced crosslinks, branching). But in dynamic aging, a more dominating chain scission prevails during the cyclic imposed aging study. The main purpose to use LAT100 specimens was to better reproduce the combined mechanical and oxidative conditions experienced by tyre tread compounds in service. Side-force data from LAT-100 testing (Figure 8d) highlights the mechanical integrity and shear resistance of the aged compounds.

For statically aged samples, side forces remain near those of unaged specimens early on and rise moderately with longer aging, reflecting slight crosslink-density growth and reduced abrasion volume. The matrix stiffens but stays cohesive enough to limit lateral deformation and material loss. Dynamic aging, by contrast, produces lower side forces and much higher abrasion volumes, showing that combined thermal–mechanical loading makes the rubber more compliant and structurally weaker.

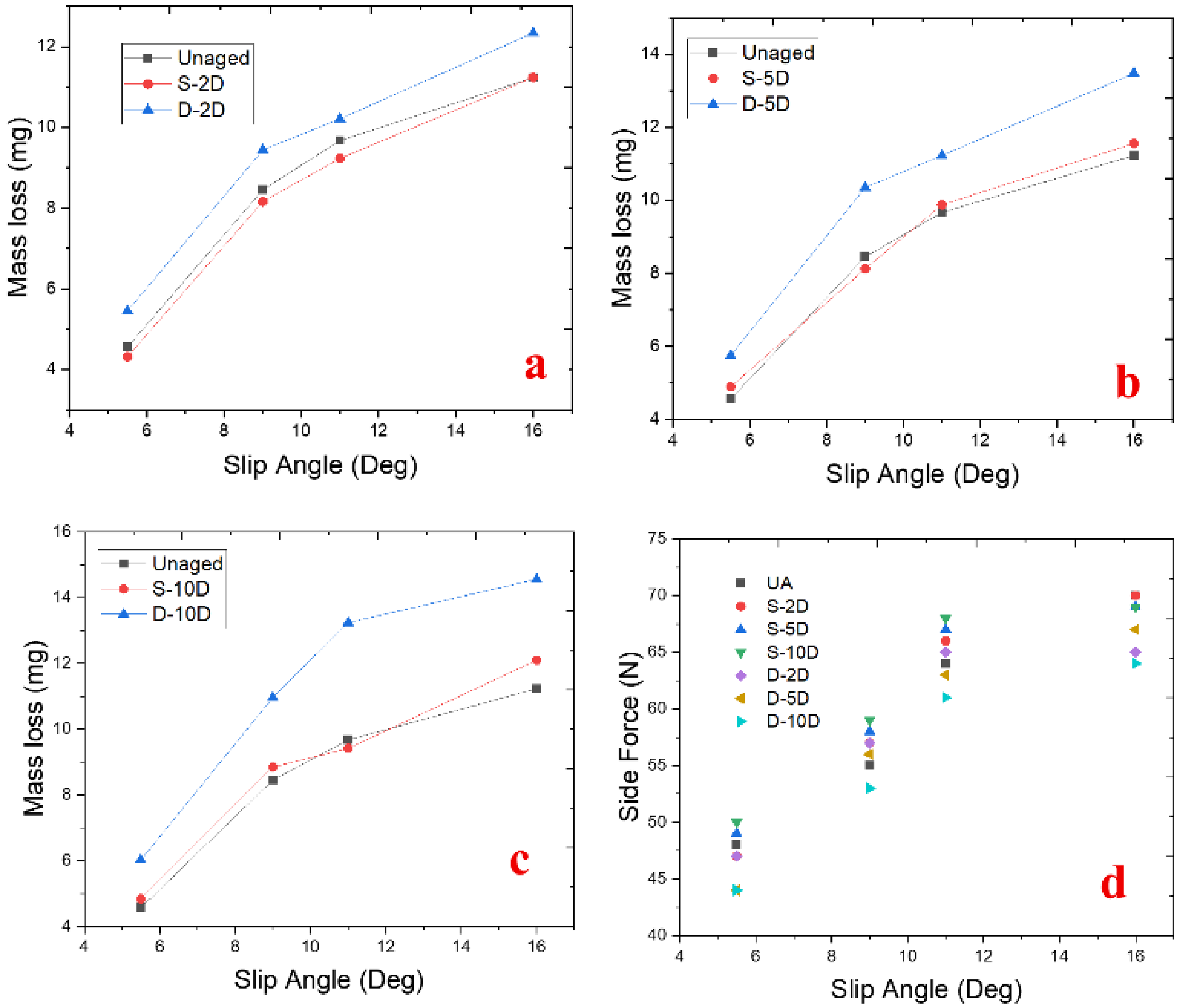

Figure 8. Effect of static and dynamic aging on the abrasion mass loss of the truck tyre compound using LAT 100 at different exposure **(a).** 2 days, **(b)** 5 days **(c)** 10 days **(d)** Side force generated during the measurement

Because it applies controlled stress during heat exposure, it replicates better in-service tire conditions than static aging. In summary, static aging primarily increases crosslink density in earlier stages, briefly improving abrasion resistance before oxidation-induced embrittlement, while dynamic aging introduces mechanical energy that accelerates radical production, oxygen proliferation, and microcrack growth. This fatigue-oxidation damage significantly reduces abrasion resistance. Dynamic aging provides enhanced sensitivity to actual degradation mechanisms and a more precise assessment of long-term tire performance degradation.

Braking efficiency was evaluated using braking force in locked-wheel LAT-100 tests, where a reduced force (N) signifies an extended braking distance (m). Figure 9 depicts results for unaged, statically aged, and dynamically aged specimens. Throughout static aging, the braking force exhibited no clear pattern throughout the 10-day duration, with relatively minimal degradation seen at maximum exposure. Conversely, dynamic aging resulted in a gradual reduction of braking force, indicating the adverse impacts of stress-induced accelerated aging. In comparing the two approaches, dynamically aged samples demonstrated significantly larger loss in braking performance, particularly after 10 days. Dynamic aging, achieved by imitating realistic road conditions by cyclic stress under heat exposure, resulted in the most significant decrease in braking force, signifying considerable degradation—cycle-loading damage, increased cracking, and decreased elastomeric resilience. Consequently, dynamic aging provides a more relevant and accurate analytical approach to assessing tire tread durability and predicting field performance.

The authors would like to make the note that the present testing protocol does not reproduce the full complexity of tyre service conditions. In particular, environmental factors such as ozone exposure, ultraviolet radiation, road contaminants, and moisture are not included.

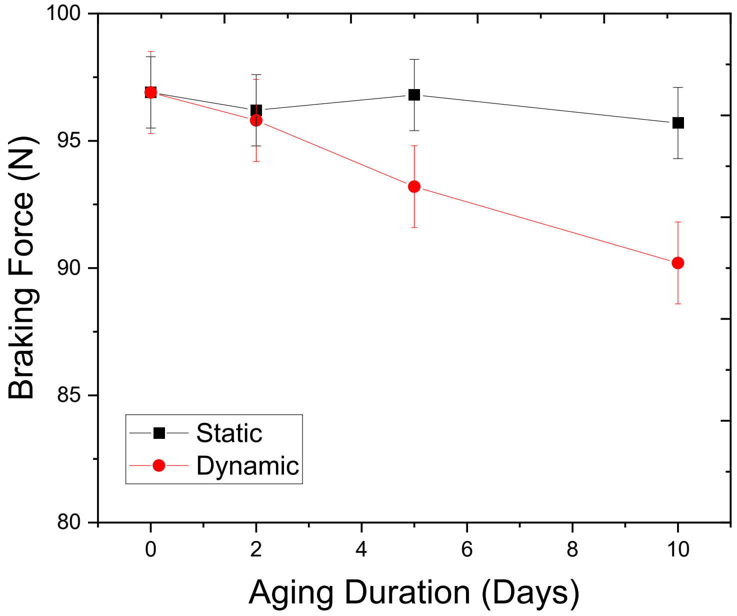

Figure 9. Effect of static and dynamic aging on the braking force using LAT 100 sample

Furthermore, the mechanical loading applied in this study represents a simplified cyclic deformation and does not capture the full multiaxial stress history experienced by tyres during actual driving conditions. Therefore, the proposed method should be viewed primarily as a laboratory-scale screening tool for comparing the dynamic thermo-oxidative stability of tread compounds and for investigating the interaction between cyclic loading and oxidative aging. Further work will be required to extend the approach by incorporating additional environmental variables and more complex loading conditions in order to better approximate real tyre service environments.

5. Conclusion

In conclusion, this study demonstrates the difference between the aging experienced by a Natural Rubber (NR) based tyre tread compound in Accelerated thermal aging (referred to as static aging) and in a cyclic stress imposed accelerated thermal aging (referred to as dynamic aging) process focusing on the network structure evolution. Mechanical behaviour was evaluated using quasi-static tensile tests, cyclic loading and Dynamic Mechanical Analysis (DMA). Key findings of the study are;

1. Increase in Stiffness with reduction in tensile strength and elongation at break of the samples was observed in both the aging modes. However, dynamic aging causes significantly higher losses in strength and resilience.
2. Dominent Stress-strain upturn is evident in Multi-step loading of the aged samples, with stress relaxation and viscoelastic responses strongly influenced by aging duration and mode; Notably larger equilibrium hysteresis loops were resulted in the stress-strain evaluation of dynamically aged specimens.
3. LAT100 evaluation reveals an increase in abrasion volume along with a decrease in braking force for both modes of aging, yet, dynamic aging produces a clearer and more progressive trend in degradation of properties. Static aging however, results in irregular abrasion and braking patterns.

The study reveals that, while static aging can offer useful insight into the long-duration oxidative degradation, it fails to capture the effect of repeated stress cycles, frictional heating, and mechanical abrasion experienced by a real tyre in service. However, the dynamic aging reproduces the operational fatigue situations of tyres, particularly in heavy loading situations typical of truck applications, where cyclical mechanical loading, thermal stress, and oxidation act in a sympathetic manner. Overall, the results confirm an advanced aging methodology for elastomeric components exposed to varying loads and an improved prediction of durability under a realistic service environment. They also highlight the importance to incorporate dynamic aging protocols in the assessment of long-term performance of rubber compounds in actual service conditions.

References

[1] J. Neethirajan, A. R. Parathodika, G.-H. Hu, and K. Naskar, Functional Rubber Composites based on Silica-Silane Reinforcement for Green Tire Application: the State of the Art. Functional Composite Materials, 3(1), (2022) 7. https://doi.org/10.1186/s42252-022-00035-7

[2] M. Sęk, W. Kaewsakul, R. Anyszka, S. Schultz, K. Bandzierz, A. Blume, Insight into the Interactions of Functionalised Rubber-Modified Silica in Tyre Tread Compounds. European Polymer Journal, 236, (2025) 114144. https://doi.org/10.1016/j.eurpolymj.2025.114144

[3] B. Fayolle, L. Audouin, G.A. George, J. Verdu, Macroscopic Heterogeneity in Stabilized Polypropylene Thermal Oxidation. Polymer Degradation and Stability, 77(3), (2002) 515-522. https://doi.org/10.1016/S0141-3910(02)00110-6

[4] Y.A. Mikheev, G.E. Zaikov, V.G. Zaikov, Heterogeneous Features of Autooxidation of Hydrocarbon Polymers. Polymer degradation and stability, 72(1), (2001)1-21. https://doi.org/10.1016/S0141-3910(00)00160-9

[5] R. Fan, Y. Zhang, C. Huang, Y. Zhang, Y. Fan, K. Sun, Effect of Crosslink Structures on Dynamic Mechanical Properties of Natural Rubber Vulcanizates under Different Aging Conditions. Journal of Applied Polymer Science, 81(3), 710-718. https://doi.org/10.1002/app.1488

[6] N. Rodriguez, L. Dorogin, K. T. Chew, B.N.J. Persson, Adhesion, Friction and Viscoelastic Properties for Non-Aged and Aged Styrene Butadiene Rubber. Tribology international, 121, (2018) 78-83. https://doi.org/10.1016/j.triboint.2018.01.037

[7] W. Di Salvi, C.E. de Santo, A. Matsumoto, A.M. Calhabeu, É.S.N. Lopes, L.P. Gabriel, Characterization of Thermal-Oxidative Aging Mechanism of Commercial Tires. Engineering Failure Analysis, 154, (2023) 107631. https://doi.org/10.1016/j.engfailanal.2023.107631

[8] M.I. Kittur, A. Andriyana, B.C. Ang, S.Y. Ch’ng, E. Verron, Inelastic Response of Thermo-Oxidatively aged carbon black filled Polychloroprene Rubber. Part II: Mullins effect. Polymer Degradation and Stability, 204, (2022)110120. https://doi.org/10.1016/j.polymdegradstab.2022.110120

[9] M.I. Kittur, A. Andriyana, B.C. Ang, S.Y. Ch’ng, E. Verron, Inelastic Response of Thermo-Oxidatively Aged Carbon Black Filled Polychloroprene Rubber. Part I: Viscoelasticity. Polymer Degradation and Stability, 205, (2022) 110118. https://doi.org/10.1016/j.polymdegradstab.2022.110118

[10] J.S. Bergström, M.C. Boyce, Large Strain Time-Dependent Behavior of Filled Elastomers. Mechanics of Materials, 32(11), (2000) 627–644. https://doi.org/10.1016/S0167-6636(00)00028-4

[11] N. Rezig, T. Bellahcene, M. Aberkane, M. Nait Abdelaziz, Thermo-Oxidative Ageing of a SBR rubber: Effects on Mechanical and Chemical Properties, Journal of Polymer Research, 27(11), (2020) 339. https://doi.org/10.1007/s10965-020-02330-y

[12] Z. Zhang, J. Sun, Y. Lai, Y. Wang, X. Liu, S. Shi, X. Chen, Effects of Thermal Aging on Uniaxial Ratcheting Behavior of Vulcanised Natural Rubber. Polymer Testing, 70, (2018).102-110. https://doi.org/10.1016/j.polymertesting.2018.06.030

[13] D.R. Bauer, J.M. Baldwin, K.R. Ellwood, Rubber Aging in tires. Part 2: Accelerated Oven Aging Tests. Polymer Degradation and Stability, 92(1), (2007)110-117. https://doi.org/10.1016/j.polymdegradstab.2006.08.014

[14] H. Wang, X. Liu, A. Varveri, H. Zhang, S. Erkens, A. Skarpas, Z. Leng, Thermal Aging Behaviors of the Waste Tire Rubber used in Bitumen Modification. Progress in Rubber, Plastics and Recycling Technology, 38(1), (2022) 56-69. https://doi.org/10.1177/14777606211038951

[15] M. Xie, H. Tang, H. Yao, Failure Analysis of Tire Separation in Two-Sized Tires on Airbus Planes. Engineering Failure Analysis, 61, (2016) 21-27. https://doi.org/10.1016/j.engfailanal.2015.07.006

[16] M. Klüppel. J. Jungk, Thermo-Oxidative Aging and Mechanical Fatigue of Elastomer Compounds Used in Various Fields of Rubber Industry. In: Heinrich, G., Kipscholl, R., Stoček, R. (eds) Degradation of Elastomers in Practice, Experiments and Modeling, Springer, Cham, Advances in Polymer Science, 289, (2022) 15–48. https://doi.org/10.1007/12_2022_114

[17] J. Neethirajan, K. MI, Thermo-Oxidative Aging and its Influence on the Performance of Silica, Carbon Black, and Silica/Carbon Black Hybrid Fillers-Filled Tire Tread Compounds. Journal of Polymer Research, 32(4), (2025) 117. https://doi.org/10.1007/s10965-025-04330-2

[18] S.C. George, M. Knörgen, S. Thomas, Effect of Nature and Extent of Crosslinking on Swelling and Mechanical Behavior of Styrene–butadiene Rubber Membranes. Journal of Membrane Science, 163(1), 1-17. https://doi.org/10.1016/S0376-7388(99)00098-8

[19] G. Kraus, Degree of Cure in Filler-Reinforced Vulcanizates by the Swelling Method. Rubber chemistry and technology, 30(3), (1957) 928-951. https://doi.org/10.5254/1.3542738

[20] G. Kraus, Swelling of Filler-Reinforced Vulcanizates. Journal of Applied Polymer Science, 7(3), (1963) 861-871. https://doi.org/10.1002/app.1963.070070306

[21] S. Polukoshko, A. Martinovs, E. Zaicevs, Experimental Studying Of Mechanical-And-Physical Properties Of Rubber During Ageing. Environment. Technology. Resources. Proceedings of the International Scientific and Practical Conference, 3, (2019) 214.

https://doi.org/10.17770/etr2019vol3.4200

[22] J.T. South, S.W. Case, K.L. Reifsnider, J.T. South, S.W. Case, K.L. Reifsnider, Effects of Thermal Aging on The Mechanical Properties of Natural Rubber. Rubber Chemistry and Technology, 76(4), (2003) 785–802. https://doi.org/10.5254/1.3547772

[23] C. Sirisinha, P. Sae-Oui, J. Guaysomboon, Mechanical Properties, Oil Resistance, and Thermal Aging Properties in Chlorinated Polyethylene/Natural Rubber Blends. Journal of Applied Polymer Science, 84(1), (2002) 22–28. https://doi.org/10.1002/app.10171

[24] P. Zhang, X. Shi, J. Li, G. Yu, and S. Zhao, Effects of Hot-Air Aging and Dynamic Fatigue on the Structure and Dynamic Viscoelastic Properties of Unfilled Natural Rubber Vulcanizates. Journal of Applied Polymer Science, 107(3), (2008)1911-1916. https://doi.org/10.1002/app.27216

[25] G. Heinrich, T.A. Vilgis, Why silica technology needs S-SBR in high performance tires?: The physics of confined polymers in filled rubbers. KGK. Kautschuk, Gummi, Kunststoffe, 61(7-8), (2008).

[26] G. Heinrich, M. Klüppel, T.A. Vilgis, Reinforced Elastomers: From Molecular Physics to Industrial Applications. Current Topics in Elastomers Research, CRC Press, (2008) 607–624.

[27] J. Plagge, M. Klüppel, Micromechanics of Stress-Softening and Hysteresis of Filler Reinforced Elastomers with Applications to Thermo-Oxidative Aging. Polymers, 12(6), (2020) 1350. https://doi.org/10.3390/polym12061350

[28] M.I. Kittur, A. Andriyana, B.C. Ang, S.Y. Ch'ng, E. Verron, Inelastic Response of Thermo-Oxidatively Aged Carbon Black Filled Polychloroprene Rubber. Part II: Mullins effect. Polymer Degradation and Stability, 204, (2022) 110120. https://doi.org/10.1016/j.polymdegradstab.2022.110120

[29] D. Besdo, J. Ihlemann, Properties of Rubberlike Materials under Large Deformations Explained by self-Organizing Linkage Patterns. International Journal of Plasticity, 19(7), (2003)1001–1018. https://doi.org/10.1016/s0749-6419(02)00090-6

[30] M. Johlitz, A. Lion, Chemo-Thermomechanical Ageing of Elastomers based on Multiphase Continuum Mechanics. Continuum Mechanics and Thermodynamics, 25(5), (2013) 605–624. https://doi.org/10.1007/s00161-012-0255-8

[31] M. Johlitz, On the Representation of Ageing Phenomena. The Journal of Adhesion, 88(7), (2012) 620–648. https://doi.org/10.1080/00218464.2012.682905

Acknowledgement

The authors thank Apollo Tyres Global R&D Asia, Chennai, India, and SRM Institute of Science and Technology, Kattankulathur, Chennai, India, for providing laboratory facilities for experimentation and characterization.

Authors Contribution Statement

Pradeep Kumar N: Conceptualization, Methodology, Project administration, Supervision, Writing - Original Draft, Writing - Review & Editing. Jeevanandham Neethirajan: Investigation, Data curation, Formal analysis, Writing - Original Draft, Writing - Review & Editing, Visualization. Rajesh Babu Ramanujam: Validation, Resources, Writing - Review & Editing. Rajendran R: Supervision, Resources, Writing - Review & Editing.

Funding

The authors declare that no funds, grants or any other support were received during the preparation of this manuscript.

Other Ethics Statements

Raw data were generated at Apollo Tyres Global R&D Asia, Chennai, India, the resource organization. Data supporting the conclusions of this study are available from the author (Pradeep Kumar N, n.pradeepkumar@apollotyres.com) upon request.

Competing Interests

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

Data Availability

The data supporting the findings of this study can be obtained from the corresponding author upon reasonable request.

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