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Dynamic Mechanical Analysis of Natural Rubber Reinforced Nanosilica Compounds in Industrial Damper Molding Machines

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A R T I C L E I N F O

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A B S T R A K

Penelitian ini mengkaji karakteristik mekanik dan redaman Karet Alam SIR 20 dengan spesifikasi teknis karet (TSR) bila dipadukan dengan komposit zeolit alam-nanosilika. Temuan menunjukkan bahwa penambahan zeolit yang dimodifikasi dengan silika amorf sebagai pengisi dalam senyawa Karet Alam TSR SIR 20 meningkatkan sifat mekanik produk vulkanisasi. Analisis Mekanik Dinamis (DMA) digunakan untuk menyelidiki sifat mekanik dan redaman. Hasilnya menunjukkan peningkatan yang signifikan pada sifat mekanik dan redaman Karet Alam yang Diperkuat dengan Senyawa Nanosilika ketika zeolit yang dimodifikasi secara magnetis digunakan. Secara khusus, rasio redaman pada sampel Si + Z melebihi sampel lainnya, melebihi ζ > 0,027. Hasil ini menunjukkan bahwa pengisi nanosilika yang diperkuat dengan zeolit alam mempunyai potensi untuk diterapkan pada perangkat peredam di industri pencetakan secara besar.

A B S T R A C T

This research examines the mechanical and damping characteristics of Natural Rubber SIR 20 with rubber technical specifications (TSR) when combined with natural zeolite-nanosilica composites. The findings show that the addition of zeolite modified with amorphous silica as a filler in the TSR SIR 20 Natural Rubber compound improves the mechanical properties of the vulcanization product. Dynamic Mechanical Analysis (DMA) is used to investigate the mechanical and damping properties. The results show a significant improvement in the mechanical and damping properties of Natural Rubber Reinforced with Nanosilica Compounds when magnetically modified zeolite is used. In particular, the damping ratio of the Si + Z sample exceeds that of other samples, exceeding ζ > 0.027. These results indicate that nanosilica fillers reinforced with natural zeolites have the potential to be applied to damping devices in considerable industry especially molding machines.

1. INTRODUCTION

A common approach to create new compounds (composites) with enhanced physical properties, such as hardness, tensile strength, and modulus, is by incorporating inorganic materials into organic polymers. Improvements have been observed even with low concentrations of these inorganic materials (Giannelis, 1998, and Ondrušová1 et al., 2018). Carbon Black (CB) is a widely used filler and stabilizer in elastomers because of its reinforcing properties. However, CB production is associated with environmental pollution as it is derived from petroleum raw materials. CB dust

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inhalation can lead to various physical and psychological health problems. Moreover, the increase of raw material cost the main raw material specially for CB production, coal tar, increasingly the rubber industry's expenses. Consequently, there is a need to explore alternative reinforcing materialsto replace CB in rubber compounds. Inorganic mineral fillers like silica, kaolin, $CaCO_3$, and zeolite (Seliem et al., 2010, and Sáenz et al., 2003) have been used to produce rubber composites. However, there have been no reports on the utilization of natural zeolite in combination with silica to create filler materials in rubber composites (Teh et al., 2004, Valadares et al., 2006, and Fang et al.,2015).

Furthermore, there is a lack of reports regarding the simultaneous application of natural materials, such as natural zeolite for modifying fillers to enhance the strength of composites containing silica (SiO_2) as a reinforcing filler in natural rubber. The use of natural materials instead of synthetic ones aligns with environmental concerns and offers several advantages, including being more eco-friendly, having significant potential, and being renewable. Aini and Indriati (2007) utilized zeolite as a paper filler to replace kaolin, and Chen et al. (2011) incorporated zeolite on the outer surface of corrugated cardboard to improve its moisture resistance and durability.

Natural zeolite is an economical and abundant absorbent material in nature (Wibowo et al., 2015).It possesses desirable properties such as ion exchange capabilities (Inglezakis et al., 2016), high adsorption capacity for inorganic and organic ions, ease of activation and regeneration, and non- toxic components (Komosiska et al., 2015). Zeolites, whether synthetic or natural, exhibit unique structural, physical, and chemical properties that make them valuable materials for various technological, agricultural, and environmental applications. The porous nature of zeolite, with its numerous cavities, enables it to absorb a wide range of polymers for the creation of functional composite materials (Dogan, 2010, and Sener, 2010). Magnetically modified zeolites offer the advantage of easy separation processes and recovery of powdered zeolites, particularly after catalytic tests or environmental cleanup, using an applied magnetic field. Magnetic zeolite applications extend to areas such as ferromagnetic ion exchange, fluidized layers of ferromagnetic particles, magnetic cooling, and biosciences (Safarik, 2002).

Indonesia currently absorbs only 16% of its total rubber production, leading to the majority beingexported abroad at lower prices. This export situation is exacerbated by the dramatic drop in raw rubber prices from $5,853 USD/ton in April 2011 to $283 USD/ton in August 2018. To address this challenge, there is a pressing need for downstream initiatives, particularly in the case of SIR 20, which accounts for over 85% of Indonesia's total natural rubber production. Leveraging SIR 20 raw materials, there is an opportunity to produce products that require excellent elasticity, such as pier bearings and other load-bearing components, including earthquake bearings. Additionally, these materials can serve as substitutes for other traditional materials, such as wooden water covers. However, the development of downstream initiatives remains limited. Therefore, there is a consideration for alternative downstream applications that can maximize the utilization of natural rubber raw materials and offer solutions to local issues.

In various applications, problems related to vibration and noise can lead to undesirable effects, such as discomforting sounds, fatigue, structural integrity concerns, reduced reliability, and decreased performance (Lv, 2006, and Sohn, 2003). Rubber is a commonly employed material for mitigating noise and controlling vibrations due to its high damping properties. It's worth noting that the loss factor is a useful parameter for evaluating the damping characteristics of rubber. Generally, a higher loss factor indicates superior damping properties.

2. METHOD

The commercial nanosilica, sourced from Brataco Chemical in Indonesia, was used as a reinforcing filler in combination with compounding materials, including white oil, ZnO, stearic acid, and sulfur, all of which were obtained from Merck. These materials were utilized without any prior treatment. Natural zeolite, originating from Sukabumi, West Java, Indonesia, underwent preparation through a ball milling process, with the particle size controlled to T45 Mesh specifications. To determine the type of zeolite used in this study, X-Ray Diffraction (XRD PW1710) was employed. The zeolite preparation method has been previously documented (Wibowo et al., 2015 and 2017). To enhance its surface area and facilitate the activation process, the zeolite was converted into a fine powder using a mechanical mortar. A specific quantity of zeolite was ground in the mortar and subsequently placed in an oven at 100 °C for 2 hours. Subsequently, the zeolite powder was transferred to an alumina crucible and heated at 600 °C under atmospheric conditions for 2 hours.

In the production of composite dampers using an accelerated sulfur curing system, the rubber formulation is outlined in Table 1. The optimization involves the utilization of white oil as a rubber softener during the mastication process and optimizing the incorporation of nanosilica, zeolite, and Fe_3O_4 fillers (a combination of all three). The compound manufacturing procedures adhere to the American Society for Testing and Materials (ASTM) - D 3184-80 standards. The composites are manufactured using a two-roll mill. Table 1 also presents the design and composition of the naturalrubber formulation employed in this study, which is consistent with previous research (Cifriadi et al., 2017 and Murniati et al., 2017). The manufacturing of the composites is carried out using the composite melting method, utilizing natural rubber SIR 20 sourced from PT Perkebunan Nusantara VIII in Bandung, Indonesia. Other chemicals used are of technical-grade quality.

Table 1. Composite formula for damping tools

Sample	Materials [phr]									
	rubber	*Oil*	ZnO	Stearic acid	Nano silica	Zeolit	Fe_3O_4	TCA	TBBS	Sulfur
Si+Z	100	10	5	2	1	3	-	1	0,7	2,25
Si+Fe	100	10	5	2	1	-	8	1	0,7	2,25
Si	100	10	5	2	1	-	-	1	0,7	2,25
Z	100	10	5	2	-	3	-	1	0,7	2,25

3. RESULT AND DISCUSSION

Clinoptilolite is a type of zeolite found in Sukabumi and has a Si/Al ratio of 4.0 to 5.0. Based on the SEM images, the average size and surface porosity are 17.66 ± 0.13 µm and 65%, respectively (Wibowo et al., 2016a and 2016b) as well as the characteristic data of zeolite used in this study as shown in Table 2, where the characterization results have been published by the author and team (Wibowo et al., 2017). The small size of the sorbent increases the surface area (m^2/g), while the high porosity of the surface increases the external surface (m^2). Natural zeolite with a highly effective surface area will have a high sorption capacity so that it will be more effective, and have good compatibility and dispersion with rubber polymers. (Ramadhan, 2015)

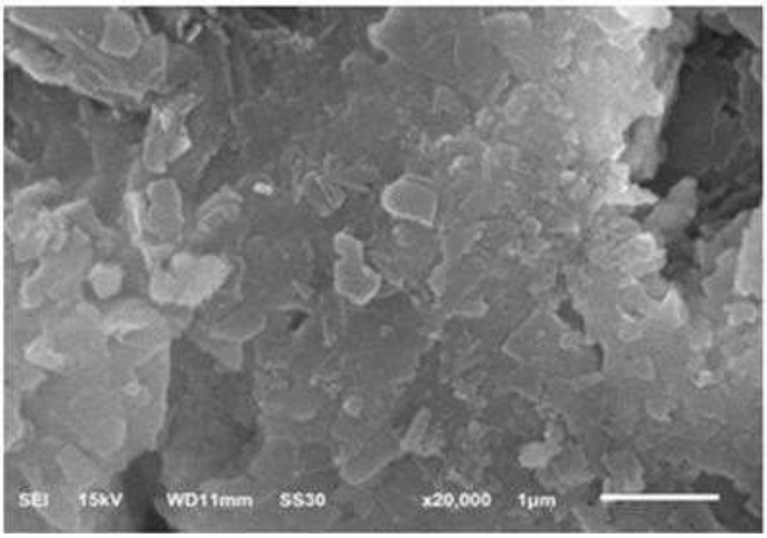

Figure 1. SEM zeolit alam

Tabel 2. Characteristics of natural zeolites

Parameters	Characteristics
Type	Clinoptilolite
Particles size	17,66 ±0,13 µm
surface specific area	33,628 $m^2.g^{-1}$
Pore volume	0,089 cm^3
Diameter pore	3,811 nm
Si/Al ratio	4.61
Thermal conductivity	0.05-6.0 W m^{-1} K^{-1}

Figure 2 shows the diffraction pattern of natural zeolite from Sukabumi. Diffraction peaks (2θ) at 9.84°, 11.14°, 13.44°, 15.31°, 17.32°, 20.97°, 22.29°, and 27.69° refer to the diffraction pattern of clinoptilolite (JCPDS 39-1383). In addition to clinoptilolite, the diffraction peaks of mordenite at 5.59°, 30.97°, 32.73°, and 34.81° (JCPDS 29-1257) and sanidine at 19.68°, 27.68° and 36.96° (JCPDS 25-0618) can also be observed. The XRD results indicate that this zeolite is not a pure clinoptilolite phase as other phases of mordenite and sanidine are included. Similar phenomena have also been reported by other authors. It has been reported that natural zeolites from Scaloma, Greece contain clinoptilolite, mordenite, cristobalite, and feldspar (Elaiopoulos, et al., 2008). Natural zeolite from Balikesir, Turkey consists of clinoptilolite, analcim, quartz, and anorthite (Yilmaz et al., 2007). To determine the degree of crystallinity of zeolites, the full width at half maximum (FWHM), crystal correlation length and lattice strain values must be determined.

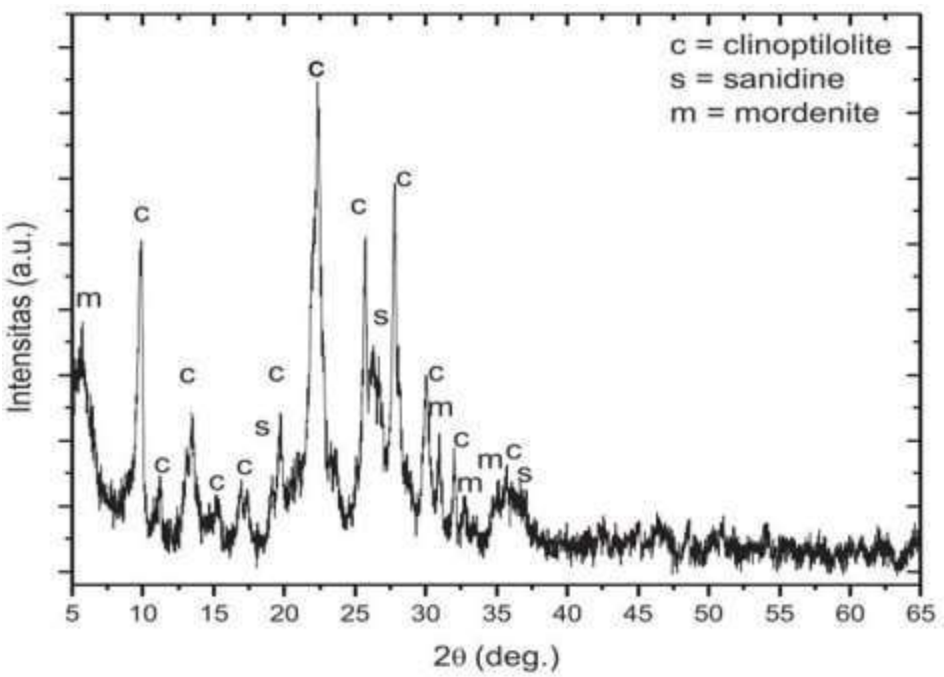

Figure 2. XRD results of natural zeolite (Wibowo et al., 2017).

Based on structural identification, we have determined that the zeolite has a monoclinic structure with a lattice spacing, d, of 3.98 Å, a crystal correlation length of 18.94 nm, a FWHM at the highest peak at 22.29° 0.85°, and a lattice strain of 9 × 10^{-3}. This natural zeolite can be considered a clinoptilolite with a high degree of crystallinity. The high degree of crystallinity of the zeolite is indicated by the small FWHM and lattice strain values. The small FWHM indicates that the zeolite has a wide crystal plane so that constructive superposition of the diffraction waves becomes easy. As a result, the resulting diffraction pattern is narrow. The small lattice strain value indicates that the atoms of the zeolite skeleton are well bound to their lattices. Therefore, zeolite crystals have homogeneous inter-lattice spacing so that the arrangement of atoms in the crystal lattice is well organized and uniformly configured. This condition causes zeolite to have a relatively high level of crystallinity.

Table 3. Chemical composition of natural zeolite

Element	Mass %	Atom %	Error %
Si	38,32	28,39	0,12
Al	7,99	6,16	0,11
Ca	3,74	1,94	0,27
K	1,36	0,27	0,22
Mg	0,92	0,79	0,10
O	47,67	62,00	0,14

The SEM micrographs in Figure 3 show the morphology of samples with silica fillers and samples with silica-zeolite mix combination fillers, and samples with unmodified and modified silica fillers. Because of the early aggregation of the silica particles, the silica-zeolite combination exhibits rather strong aggregation in both the photos with and without softeners. On the other hand, it was discovered that the magnetically altered zeolite in the silica particles was comparatively welldistributed, had no aggregation, and an average particle size of between 300 and 150 nm. Some of the particles were comparatively poorly spread, and fissures were seen in both of the Si filler photos.

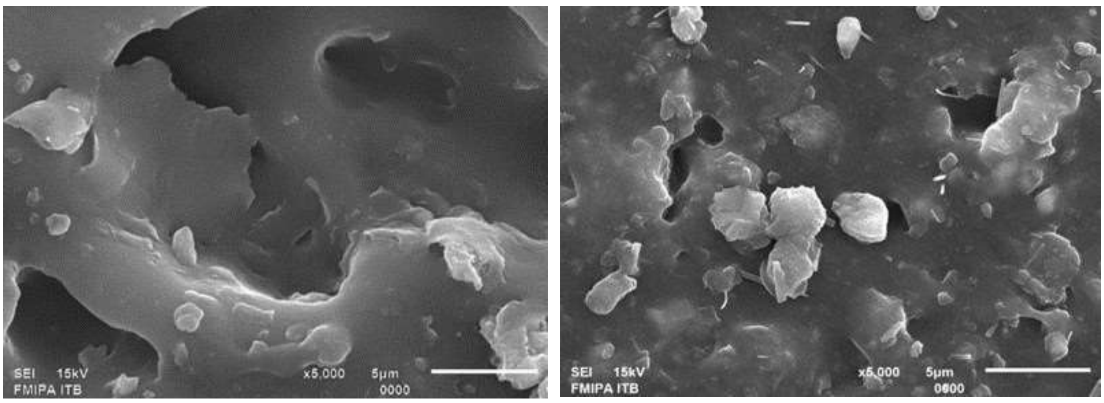

Figure 3. SEM images of samples with Si filler (left) and samples with silica-zeolite filler (right)

Reference mechanical properties of high-quality damping rubber in 2 mm thick and 23^0C temperature test specimens provided by Tun Abdul Razak Research Center (UK) as shown in Table 4 Damping solutions used in engineering settings may require a high damping coefficient, which means that instead of returning energy to the system, unwanted vibrations will be 'absorbed' by the materialand reduce any response.

Tabel 4. Properties of rubber dampers

Properties (source: TARRC)	**HDR** (High Damping Rubber)
Bulk Modulus (MPa)	2500
Tensile Strength (MPa)	13,5
Elongation at Break (%)	700
Shear Modulus (MPa)	0,55
Damping (%)	14
At T=23 C, thickness 2 mm, f = 0.5 Hz	

Stress and strain parameters are defined, but the main parameters in either frequency and temperature sweep tests are temperature, storage modulus, and loss modulus. Stress in phase with strain in sinusoidal shear deformation divided by strain is the definition of dynamic storage modulus (E') (An, et al., 2011). Figure 3 shows the loss factor relationship for the composites under study as a function of temperature.

When the strain is divided by the strain, the stress coming out of the 90° phase is known as the lossmodulus (E"). It is a measurement of the energy lost as heat during each cycle of deformations, or viscous reaction of the material (An, et al., 2011). The fluctuations of E" and E' for the various composites at a temperature of 23 °C and a frequency of 10 Hz are shown in Figure 4. These results clearly show that the loss modulus peak expands with the addition of filler. this could be explained by the fact that the addition of filler causes the relaxation process in the composite to be inhibited due to the increase in the number of chain segments (Wode, et al., 2012). Due to the increased molecular mobility of the polymer chains, E' decreases for all composites as the temperature increases (Rahmanian, et al., 2013); conversely, at temperatures above 90°, the storage modulus value increases.

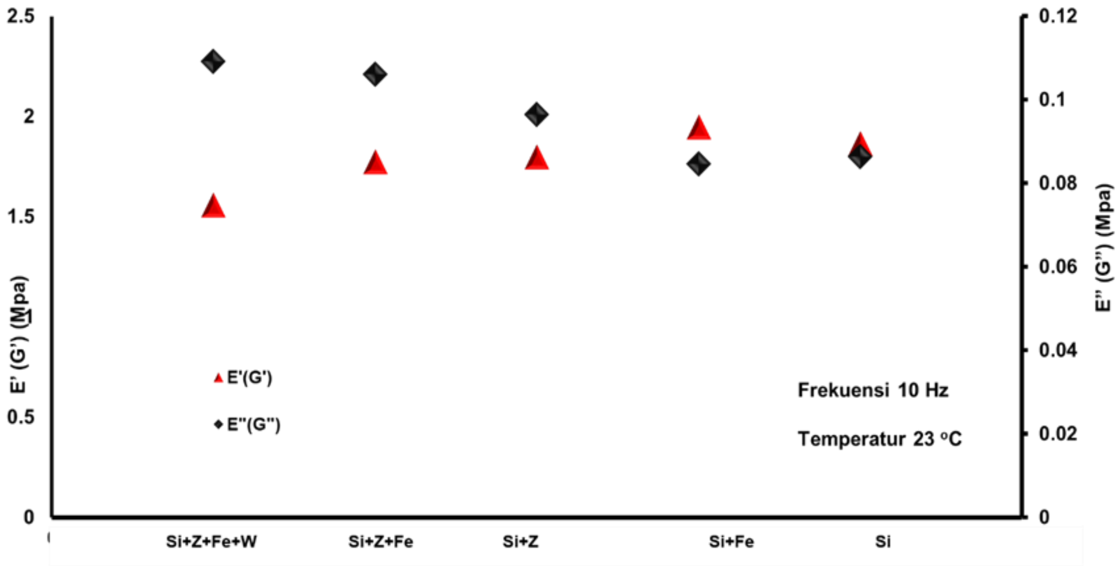

Figure 4. Storage modulus (E') and loss modulus (E")

The most exciting information observed in Figure 5 are the Si+Z decay curves at frequencies of 0.1 Hz, 1 Hz, and 10 Hz. The other samples have similar decay curves, typical of general solutions for damped vibrations, with varying deformation parameters and different decay times. Assuming the vibration obeys linear properties, we have a general solution for damped vibration as

$$y = Ae^{-\gamma t}\sin(2\pi f t + \emptyset) \tag{1}$$

with

$$\gamma = \omega_n \zeta \tag{2}$$

where ζ is the damping ratio, ζ = η/2, η is the loss factor (tan θ), f = p/2 and A is the amplitude ofeachsample at each frequency (f) that we get from the strain data of the DMA results. The exponential decay equation (Sun and Bai, 1995) makes the system oscillate with exponentially decreasing amplitude.

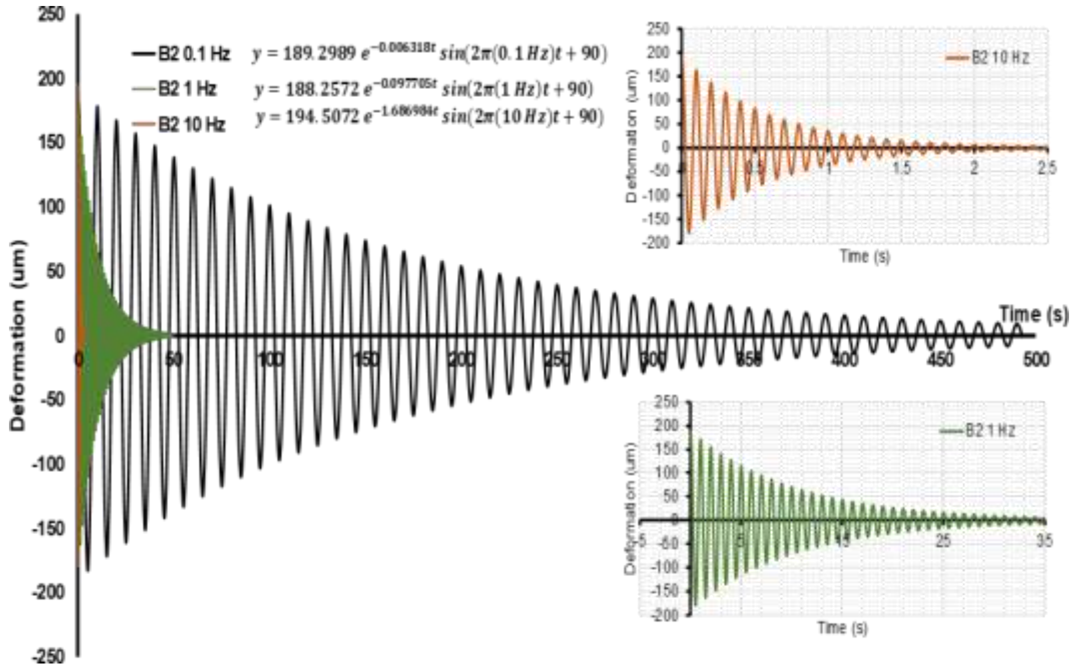

Figure 5. Decay curve of the Si+Z sample

Damping causes the amplitude of the oscillation to gradually decrease over time, and how fast it reduces depends on the damping factor. Reviews of loss factors in different damping materialsat 1 Hz are in the range of 0.039-0.8 (Wode et al., 2012), and this experiment yielded loss factor values in this range with damped decay curves of all samples with frequencies of 0.1 Hz, 1 Hz, and10 Hz.

The filler in the Si+Z+Fe+W has better damping and mechanical properties because the magnetically modified zeolite helps the amorphous silica become a better-reinforcing filler, we can see a clear comparison from other samples. In fact, the loss factor of rubber insulators is highly dependent on the frequency of vibration and noise, and rubber insulators will not be able to adapt tothe changing vibration and noise frequencies (Lejon and Kari, 2009), tan δ raw data was measuredat lower frequency levels (less than 20 Hz)..

4. CONCLUSION

Dynamic Mechanical Analysis (DMA) was employed to examine the mechanical and damping properties of the materials. The findings indicate a substantial enhancement in the mechanical and damping characteristics of Natural Rubber Reinforced with Nanosilica Compounds when magnetically modified zeolite is incorporated. Notably, the damping ratio of the Si + Z sample surpasses that of the other samples, exceeding ζ > 0.027. These results suggest that the use of nanosilica fillers reinforced with natural zeolites holds promise for applications in damping devices within various industrial sectors especially molding machines.

5. ACKNOWLEDGE

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