



Optimizing EPDM/Silica Composites for High-Pressure Hydrogen Sealing Applications: Role of Silica Characteristics and Content

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Abstract: This study evaluates the effects of silica surface area and loading on the hydrogen resistance and mechanical stability of ethylene propylene diene rubber (EPDM)/silica composites for advanced sealing applications. EPDM was compounded with silica grades ranging from 55 to 225 m²/g at 30, 50, and 70 phr. After peroxide curing, the specimens were exposed to 96.3 MPa hydrogen at room temperature for 24 h. Properties including hydrogen permeability, swelling, mechanical strength, dynamic modulus, and hysteresis were examined. The composite containing 50 phr of high-surface-area silica (225 m²/g) exhibited the optimal balance of low permeability, reduced swelling, and stable mechanical response. By contrast, 70 phr loading caused aggregation and strength loss. These results indicate that optimizing the filler surface area and content is critical in designing EPDM composites for hydrogen-sealing applications.

Keywords: nanocomposites, high-pressure hydrogen exposure, hysteresis loss, dynamic mechanical analysis, chain mobility

Introduction

Hydrogen is currently attracting attention as a clean energy source, and the rapid growth of the automobile industry has accelerated its use in fuel cell vehicles. With the expansion of hydrogen mobility, the reliability of the charging process and the safety of related components have become critical issues. In particular, high-pressure hydrogen charging systems require sealing materials that can maintain performance under severe conditions.¹⁻⁵

Elastomeric rubbers are applied for this purpose, but they are prone to hydrogen-induced degradation that can compromise sealing integrity. Ethylene-propylene-diene monomer (EPDM) rubber is one of the most common candidates because of its excellent weather resistance, ozone stability, and flexibility across a wide temperature range.⁶⁻⁸ However, its relatively high hydrogen permeability and susceptibility to hydrogen-related microstructural damage limit long-term reliability in hydrogen service.

Mechanical deterioration of rubber seals under high-pressure hydrogen is often associated with internal cracking,

or blister fracture, which reduces durability. Previous investigations have shown that hydrogen exposure markedly affects EPDM behavior. Yamabe et al.⁹⁻¹² and Koga et al.^{13,14} demonstrated that higher hydrogen pressures and elevated temperatures accelerate blister formation in unfilled EPDM. In addition, Yamabe et al. reported that repeated hydrogen exposure, especially under compression-decompression cycling, promotes internal cracks, with the decompression rate, pressure, and temperature playing key roles.

To enhance the durability of rubber materials in harsh environments, the incorporation of reinforcing fillers such as carbon black, organoclay, and silica has been extensively studied.¹⁶⁻²⁰ Yamabe et al.^{21,22} and Nishimura⁵ investigated the effect of the type and amount of filler on hydrogen permeation and blister fracture behavior following exposure to 10 MPa hydrogen gas. They observed that increasing the filler content reduced the severity of blister fractures in EPDM vulcanizates, attributed to the reinforcing effect of the fillers. Moreover, when comparing silica- and carbon black-filled EPDM, the silica-filled composites absorbed significantly less hydrogen than those containing carbon black.

In this study, the influence of silica surface area and loading level on the hydrogen durability of EPDM/silica

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nanocomposites was investigated. Precipitated silicas with surface areas ranging from 55 to 225 m²/g were incorporated into EPDM at 30, 50, and 70 phr and crosslinked through a peroxide system. The cured specimens were subjected to high-pressure hydrogen (96.3 MPa, room temperature, 24 h), and their swelling behavior, gas permeability, rheology, dynamic mechanical properties, and hysteresis were examined. The aim of this study is to clarify how filler characteristics affect structural integrity and interfacial stability, and to identify conditions that provide reliable sealing performance in hydrogen service environments.

Experimental

1. Sample Preparation

Ethylene propylene diene rubber (EPDM, NORDEL IP 4760P, ethylene content: 67.5%, ethylenenorbornene content: 5%) was procured from Dow Chemical Co., USA. Precipitated silica (Ultrasil 360, Ultrasil VN2, Ultrasil 7000GR, Ultrasil 9100GR) with the BET area of 55, 130, 175 and 225 m²/g was supplied by Evonik, Germany. As a silane coupling agent, Bis-(γ -triethoxysilylpropyl)tetrasulfide (TESPT, Nanjing Aocheng Chemical Ltd., China) was used. Polyethylene glycol (PEG-4000) was purchased from Green Chemical Ltd., Korea. Dicumyl peroxide (DCP, Perkadox BC-FF) was supplied by Nouryon, Netherlands. Triallyl cyanurate (TAC) was procured from Hunan Farida

Technology Co., China. Lastly, stearic acid and zinc oxide were purchased from PJ CHEMTEK Ltd., Korea, and LG Household & Health Care, Korea.

The EPDM/silica composites were compounded with different silica contents (0, 30, 50, and 70 phr). The detailed formulation of the composites is shown in Table 1. For simplicity, the EPDM/silica composites were named Sx-y, where x and y indicate the BET area and the filler's phr content of the silica, respectively. For example, S55-70 is an EPDM/silica composite filled with 70 phr of silica, with a BET area of 55 m²/g.

The rubber vulcanizates were prepared using a two-step mixing process. In the first step, EPDM rubber was masticated and then blended with precipitated silica, silane coupling agent, and processing aids in an internal mixer (3L kneader, Moriyama Co., Japan). Mixing was carried out at an initial temperature of 85°C, reaching a dump temperature of 140°C, with the rotor set to 30 rpm for about 30 minutes. In the second step, the curing agent and co-agent were incorporated into the compound using an 8-inch two-roll mill (PK-RM20140930, Pungkwang Co., Korea). The final compounds were then vulcanized by compression molding at 160°C, with the molding time set to t_{90} , as determined by a moving die rheometer (MDR).

2. Characterization

Curing behavior was analyzed using the MDR according

Table 1. Formulation of the EPDM/Silica Composites

[phr]	Neat	S55-70	S130-70	S175-70	S225-30	S225-50	S225-70
EPDM				100			
Silica	0	70	70	70	30	50	70
Stearic acid				1.0			
Zinc oxide				3.0			
PEG-4000	0	2.8	2.8	2.8	1.2	2.0	2.8
TEPST	0	5.6	5.6	5.6	2.4	4.0	5.6
DCP				1.5			
TAC				1.0			

Table 2. Hysteresis Loss and Shear Modulus of EPDM/Silica Composite by Specific Surface Area

		Neat	S55-70	S130-70	S175-70	S225-70
Unexposed	Hysteresis loss [%]	14.7	39.8	42.3	43.3	45.8
	Shear modulus [kgf/cm ²]	85.0	172.5	191.5	194.5	239.5
Exposed	Hysteresis loss [%]	15.3	40.2	41.1	44.0	46.3
	Shear modulus [kgf/cm ²]	81.8	187.4	197.1	227.8	257.6

to the ASTM D2084 standard at 160°C. The oscillation frequency was 100 cycles/min⁻¹ with an amplitude of 1°.

The hardness was measured according to ASTM D2240 with Shore A durometer (ASKER Type A, KOBUNSHI KEIKI Co. Ltd., Japan). The mechanical properties were evaluated using a universal testing machine (UTM, Daekyung, Korea) according to the test method A in ASTM D412 at room temperature. The crosshead speed was 500 mm/min, and at least 5 samples were tested for the accuracy of the data.

Hydrogen permeability of the filled EPDM vulcanizates was evaluated using high-purity hydrogen gas (99.9999%) and the differential pressure technique. Disc-shaped specimens (45 mm in diameter, 2 mm in thickness) were mounted between an upper and lower chamber, with a porous support ensuring a constant effective permeation area. Initially, the lower chamber was evacuated to 0.001 kPa, followed by the upper chamber to 0.1 kPa. Hydrogen gas was then introduced into the upper chamber until it reached 100 kPa. The pressure increase in the lower chamber was monitored until it reached 1.3 kPa, indicating hydrogen permeation through the specimen. From the resulting pressure profile, the hydrogen permeability coefficient (P), diffusivity (D), and solubility (S) were determined using the following equation.²³

$$S = \frac{P}{D}$$

High-pressure hydrogen exposure was performed in a cylindrical chamber using 99.999% pure hydrogen gas at room temperature. Before pressurization, the system was purged three times with hydrogen gas at 5 MPa to eliminate residual atmospheric gases. Disc-shaped specimens with a thickness of 2 mm and a diameter of 15 mm were then placed in the chamber, which was pressurized at a rate of approximately 5 MPa/min until reaching 96.3 MPa. The samples were maintained under this condition for 24 hours to ensure sufficient hydrogen sorption. Decompression was carried out rapidly at a rate of approximately 1 MPa/s. Immediately after pressure release, the samples were transferred to a measurement setup in which desorbed hydrogen displaced the liquid in a graduated cylinder. The change in liquid level was recorded and used to calculate the amount of absorbed hydrogen using a volumetric analysis method.^{24,25}

Material volume change after exposure to high-pressure hydrogen gas (96.3 MPa) was measured with a disc-type specimen of 45 mm diameter and 2 mm thickness. The samples were exposed for 24 hours at room temperature.

The thickness and diameter of the specimens were measured before and immediately after high-pressure hydrogen exposure, and the volume change was calculated based on the dimensional differences. The volume change values were obtained by following the equation:

$$\Delta V = \frac{V_f - V_i}{V_i} \times 100(\%)$$

where V_f is the volume of the sample after decompression and V_i is the volume before exposure to hydrogen gas.

Changes in tensile strength were evaluated after exposing the specimens to high-pressure hydrogen gas at 96.3 MPa for 24 hours at ambient temperature. The samples used for the test were according to ASTM D412. The tensile strength changes of before and immediately after exposure to hydrogen were calculated using the following equation:

$$\Delta M = \frac{M_A - M_B}{M_B} \times 100(\%)$$

where M_A is the mechanical properties after decompression, and M_B is the initial tensile strength before exposure to a hydrogen environment.

A volume swelling test was performed to obtain the crosslink density of the composites. A rectangular-shaped sample with dimensions of 10 × 10 × 2 mm³ was put into tetrahydrofuran (THF) for 72 h at room temperature and then removed. After cleaning the excess THF from the surface of the samples, the swollen samples were weighed. The volume swelling ratio (V_Q) was determined from the weight of the sample in the unswollen and swollen states using the following equation:

$$V_Q = \frac{(W_s - W_d)/\rho_s}{W_d/\rho_r} \times 100(\%)$$

V_Q is the volume swelling ratio, W_d is the initial weight of the sample, W_s is the weight of the sample in the swollen state, ρ_r is the density of the EPDM, and ρ_s is the density of the THF.

The crosslink density was calculated according to the Flory-Rehner equation as follows:

$$V = \frac{1}{2M_c} = -\frac{\ln(1 - V_1) + V_1 + \chi V_1^2}{2\rho_r V_0 \left(V_1^{\frac{1}{3}} - \frac{V_1}{2} \right)}$$

$$V_1 = \frac{\frac{W_d - W_f}{\rho_r}}{\frac{W_d - W_f}{\rho_r} + \frac{W_s - W_d}{\rho_s}}$$

where V is the crosslink density (mol/g), M_c is the average molecular weight between crosslink points (g/mol), V_0 is the molar volume of solvent (cm³/mol), V_1 is the volume fraction of rubber in the swollen gel at equilibrium, W_d is the weight of the unswollen sample, W_f is the weight of the filler in the sample, W_s is the weight of the swollen sample, ρ_r is the density of EPDM, ρ_s is the density of THF, and χ is the polymer-solvent interaction parameter ($\chi = 0.501$).¹⁹

Hysteresis loss was measured using a cyclic tension test on a UTM (Daekyung, Korea). A preconditioning test was done before the actual test to minimize the Mullins effect. The test conditions were set to a crosshead speed of 500 mm/min, an extension length of 20 mm (strain of 100%).

The dynamic mechanical analysis was performed using a dynamic mechanical analyzer (ARES G2, TA Instruments, USA). The test strain range was from 0.01 to 100%.

The samples were subjected to a frequency of 1 Hz at a temperature of 25°C. The results are presented in terms of storage modulus and $\tan \delta$ as a function of strain.

Lastly, the fractured surfaces of the composites were observed by field-emission scanning electron microscopy (FE-SEM, JSM-6710F, JEOL Ltd., Japan) at a voltage of 15 kV under an N₂ atmosphere. After 24 hours of exposure to a high-pressure hydrogen environment, the surface specimens were prepared and sputter-coated with gold.

Results and Discussions

1. Curing Behavior and Crosslink Density Evolution

The incorporation of silica into EPDM enhanced the curing behavior by increasing torque development during

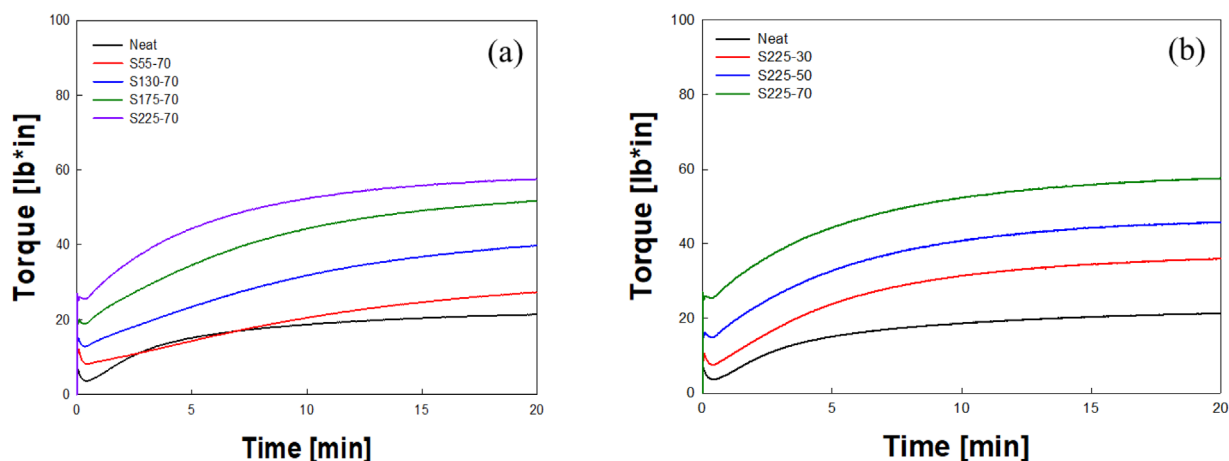


Figure 1. Rheo curve of EPDM/silica composites: (a) by specific surface area and (b) by silica content.

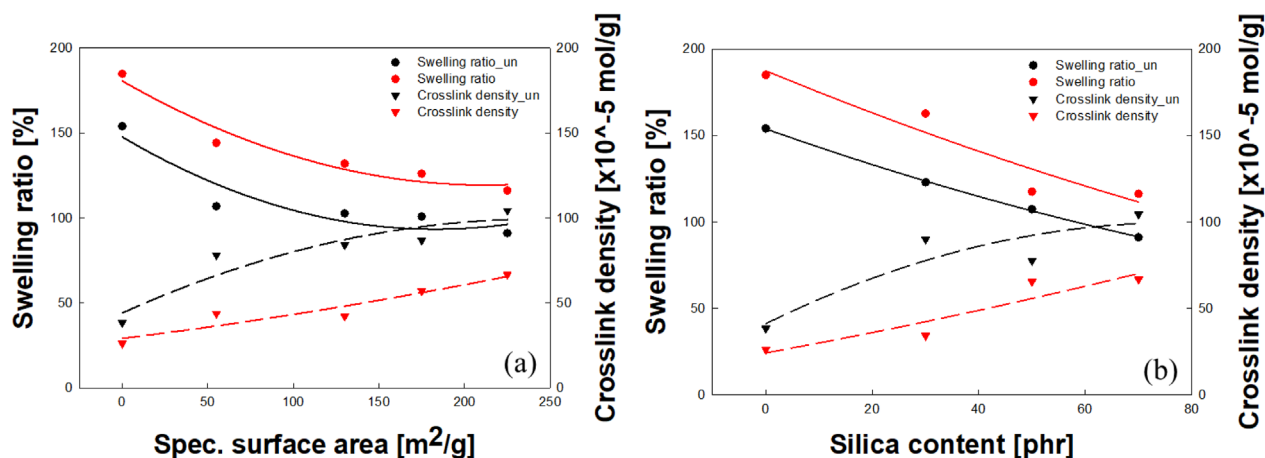


Figure 2. Swelling ratio and crosslink density of EPDM silica composites after exposure to hydrogen: (a) by specific surface area and (b) by silica content.

Table 3. Hysteresis Loss and Shear Modulus of EPDM/Silica Composite by Silica Content

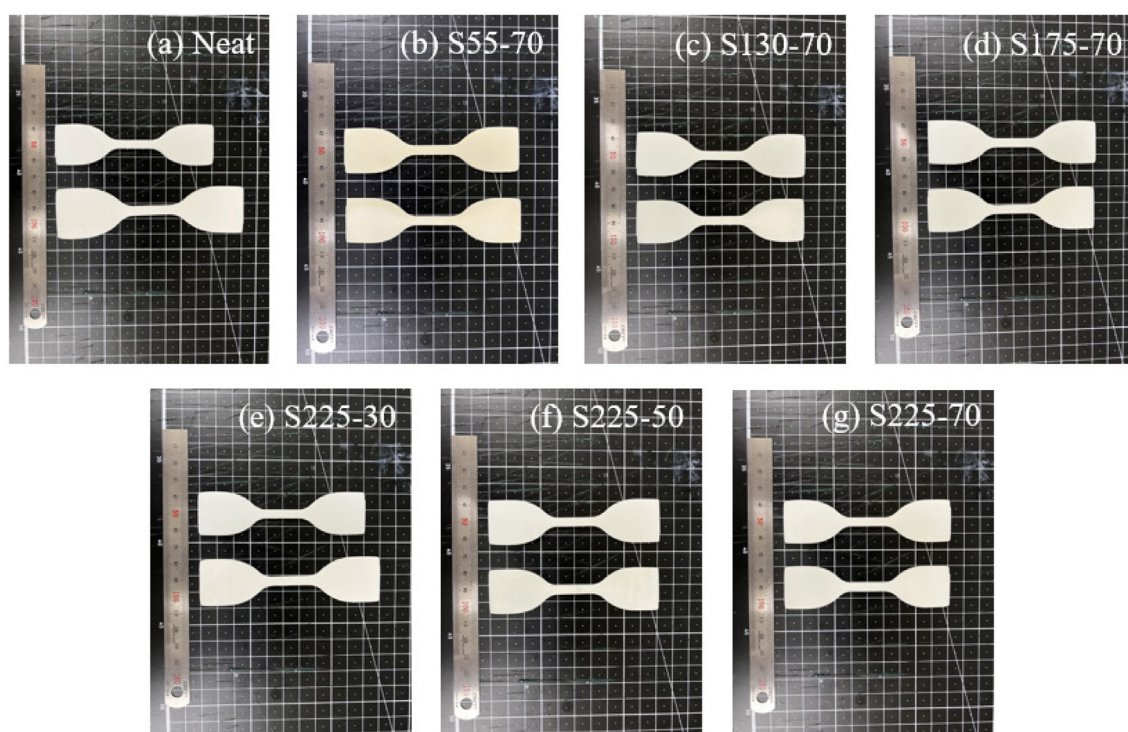
		Neat	S225-30	S225-50	S225-70
Unexposed	Hysteresis loss [%]	14.7	29.2	36.9	45.8
	Shear modulus [kgf/cm ²]	85.0	138.0	187.0	239.5
Exposed	Hysteresis loss [%]	15.3	29.1	38.1	46.3
	Shear modulus [kgf/cm ²]	81.8	140.9	215.9	257.6

vulcanization, reflecting higher crosslink density formation (Figure 1). High surface area silica provided abundant active sites for polymer-filler interactions, thereby forming a more rigid network. As silica loading increased, torque values rose progressively. However, excessive filler content risked particle agglomeration and localized stress concentration (Figure 1b). Hydrogen exposure reduced crosslink density in all systems, with low surface area silica showing the most severe reduction. The swelling ratio and calculated crosslink density results confirmed that high surface area silica was more effective in maintaining network integrity under hydrogen environments (Figure 2).

2. Hydrogen Permeation and Absorption Characteristics

The addition of silica was found to reduce hydrogen

permeability by restricting free volume and generating tortuous diffusion pathways, as shown in Figure 4. However, barrier improvement was not proportional to loading. While moderate filler contents reduced permeability, higher loadings led to plateau or even slight increases due to filler aggregation and the formation of preferential diffusion channels, particularly in the S225 series. High surface area silica demonstrated respectively superior barrier performance at equivalent loadings by achieving better dispersion and more restricted chain mobility. A similar effect was observed for hydrogen absorption: neat EPDM exhibited the highest residual hydrogen content (2303 ppm), whereas silica-filled composites showed markedly reduced values (Figure 5). The lowest residual hydrogen was demonstrated in the S225-70 formulation (85.98 ppm), suggesting that an optimal balance of crosslink density and filler dispersion enhances hydrogen

**Figure 3.** Images of the samples before and after exposure to high-pressure hydrogen.

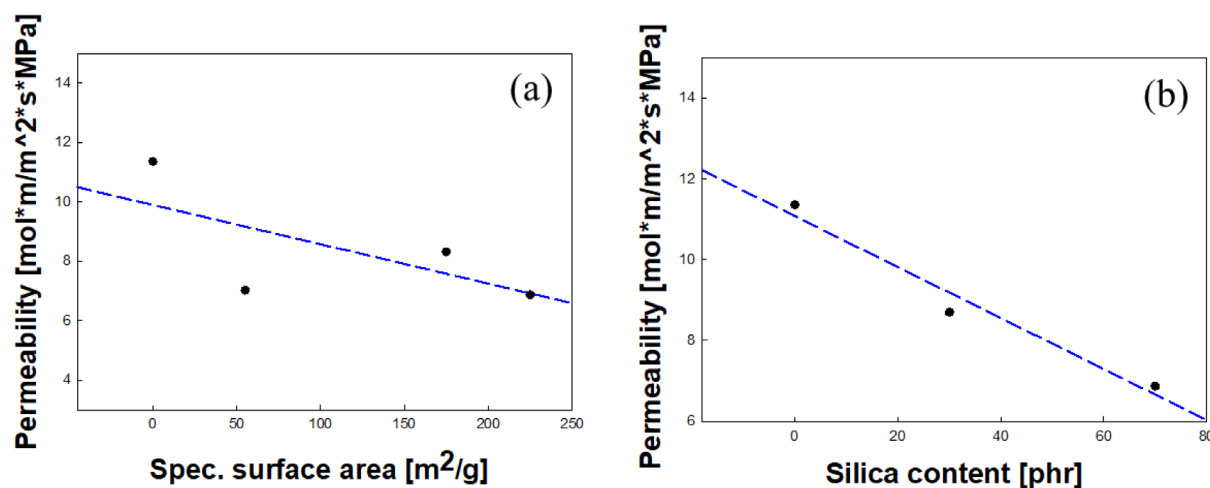


Figure 4. Permeability of EPDM/silica composites before & after hydrogen exposure: (a) by specific surface area and (b) by silica content.

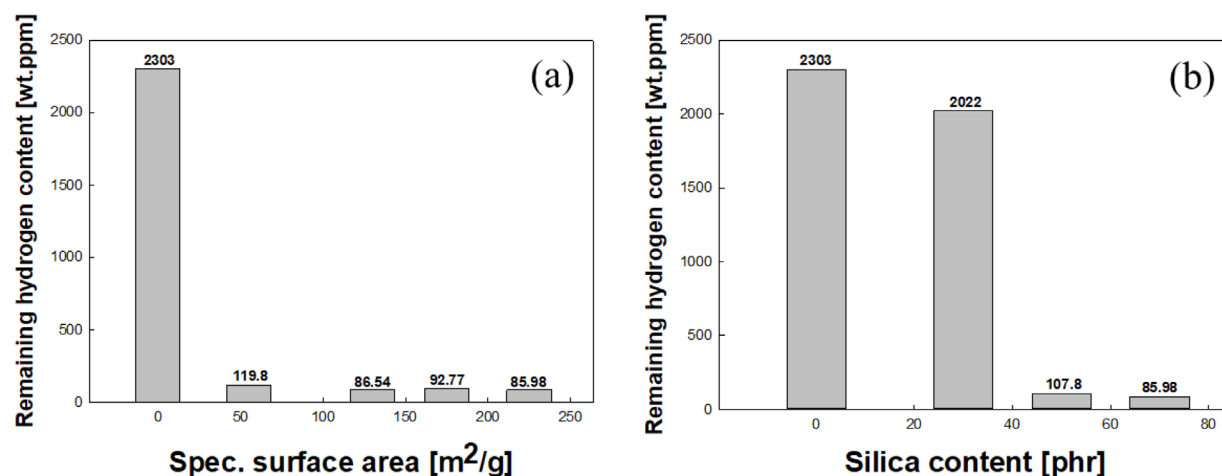


Figure 5. Remaining hydrogen content of EPDM/silica composites before & after hydrogen exposure: (a) by specific surface area and (b) by silica content.

release. These results confirm that surface area-dependent interactions at the polymer-filler interface play a decisive role in reducing hydrogen solubility.

3. Mechanical Property Retention under Hydrogen Exposure

Silica reinforcement improved the tensile strength of EPDM, with higher surface area and increased loading leading to greater improvements prior to hydrogen exposure (Figure 6). This effect arises from enhanced interfacial interactions, which promote more efficient stress transfer across the matrix. After hydrogen exposure, tensile strength decreased slightly across all samples, but the reduction was most pronounced at the highest filler content (70 phr).

The results indicate that excessive loading may deteriorate filler-matrix adhesion and promote micro-void formation. Stress-strain analysis further supported this conclusion: neat EPDM exhibited reduced tensile strength but retained elongation, pointing to crosslink degradation as the dominant mechanism, whereas composites with high surface area silica, especially S225-50, maintained both strength and elasticity (Figure 7). These findings demonstrate that well-dispersed silica networks enhance resistance to hydrogen-induced degradation, whereas agglomerated fillers reduce this benefit.

4. Dimensional Stability of Composites

Neat EPDM underwent substantial swelling under high-pressure hydrogen, with volume expansion reaching approxi-

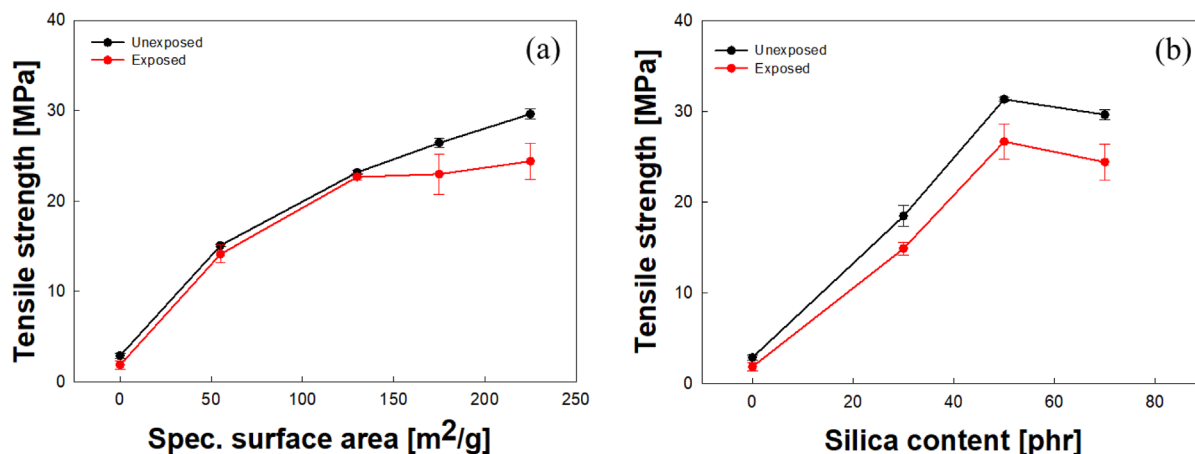


Figure 6. Tensile strength changes of EPDM/silica composites before and after hydrogen exposure: (a) by specific surface area and (b) by silica content.

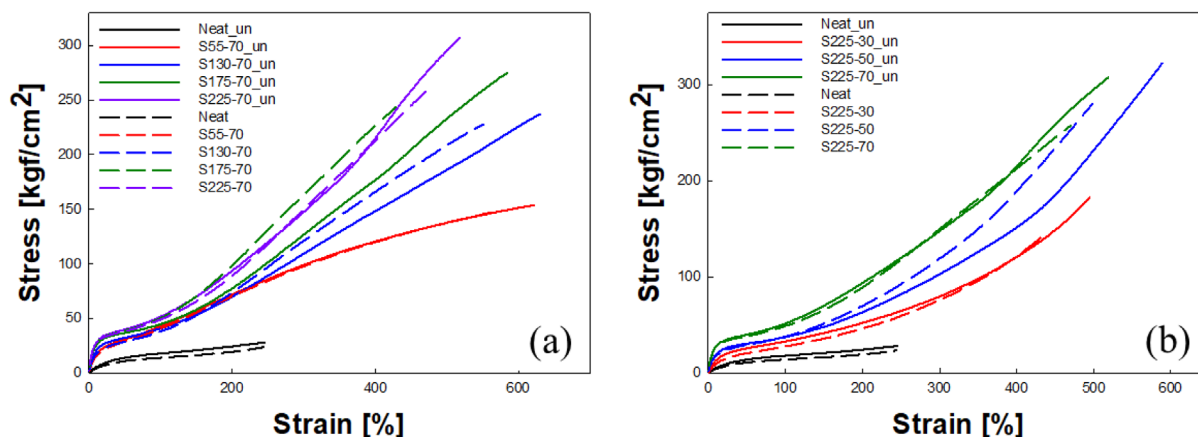


Figure 7. S-S curve of EPDM/silica composites before and after hydrogen exposure: (a) by specific surface area and (b) by silica content.

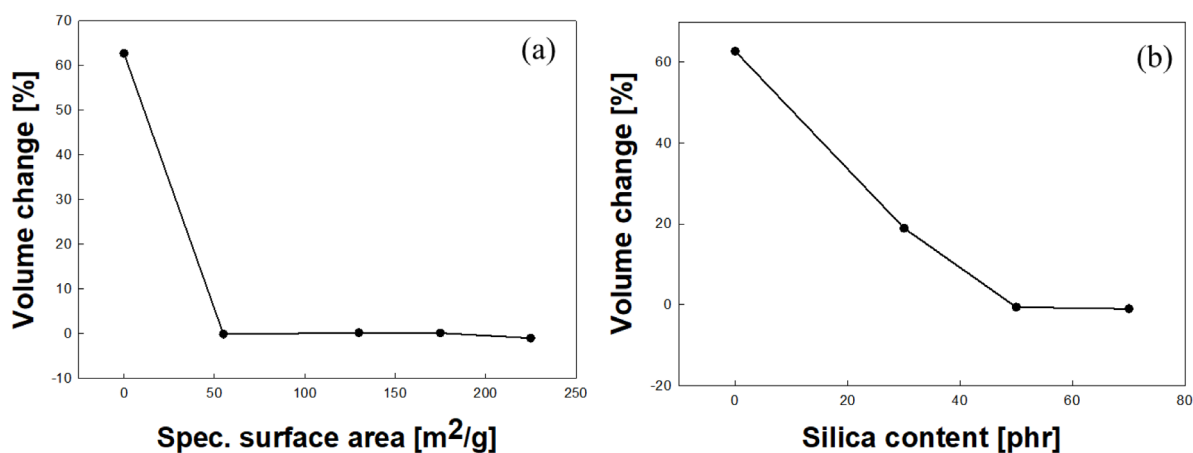


Figure 8. Volume change of EPDM/silica composites before and after hydrogen exposure: (a) by specific surface area and (b) by silica content.

mately 60% (Figure 8). Silica incorporation markedly suppressed this dimensional change by constraining polymer

chain mobility and reinforcing the network structure. The improvement was most notable for high surface area silica,

which exhibited the lowest swelling ratio. The crosslink density data (Figure 2) corroborated these observations, showing that higher filler-polymer interactions reduce chain mobility and suppress hydrogen-induced expansion. However, while the

S225-70 formulation achieved superior dimensional stability, excessive filler loading simultaneously led to increased mechanical deterioration, highlighting the trade-off between swelling suppression and structural resilience.

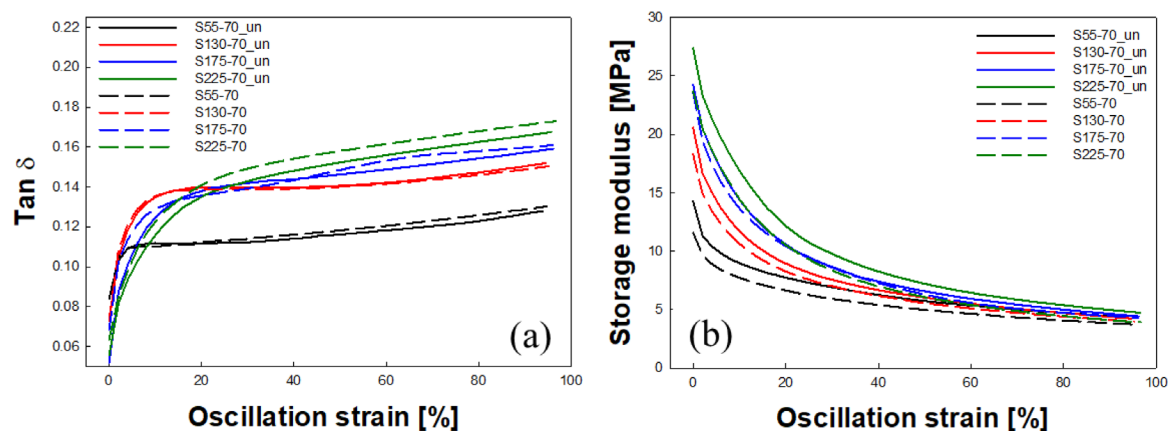


Figure 9. Dynamic mechanical analysis result of EPDM/silica composites by specific surface area: (a) Tan δ and (b) storage modulus.

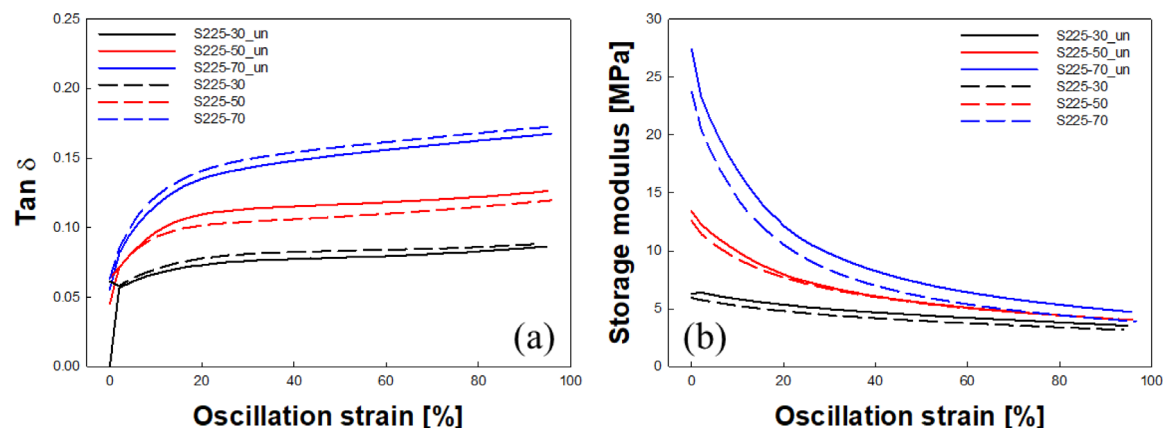


Figure 10. Dynamic mechanical analysis result of EPDM/silica composites by silica content: (a) Tan δ and (b) storage modulus.

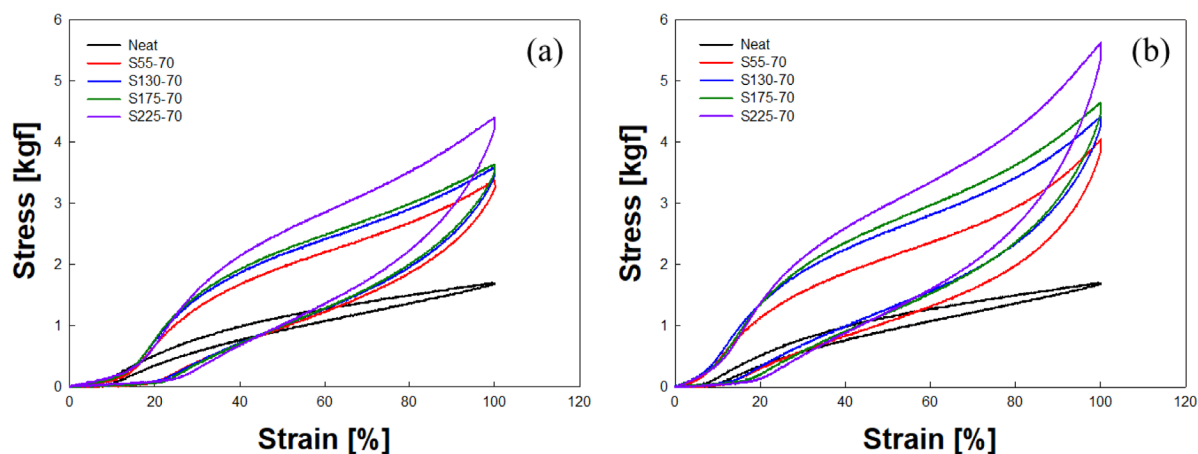


Figure 11. Hysteresis loss of EPDM/silica composite by specific surface area: (a) Before H₂ and (b) after H₂ exposure.

5. Viscoelastic Response and Hysteresis Behavior

Dynamic mechanical analysis revealed that hydrogen exposure increased $\tan \delta$ values, particularly at higher strains, indicating greater energy dissipation due to network disruption and enhanced chain mobility (Figure 9). Storage modulus (E') measurements further showed nonlinear softening under hydrogen exposure, with high surface area

silica composites retaining modulus more effectively than those with lower surface area (Figure 10). Cyclic tension test confirmed these trends: hydrogen exposure increased hysteresis loss, especially in highly filled composites, as a result of filler network disruption and chain scission (Figure 11, Figure 12). In contrast, S225-based composites, particularly at 50 phr, displayed lower energy loss and more stable mechanical response. These results indicate that high

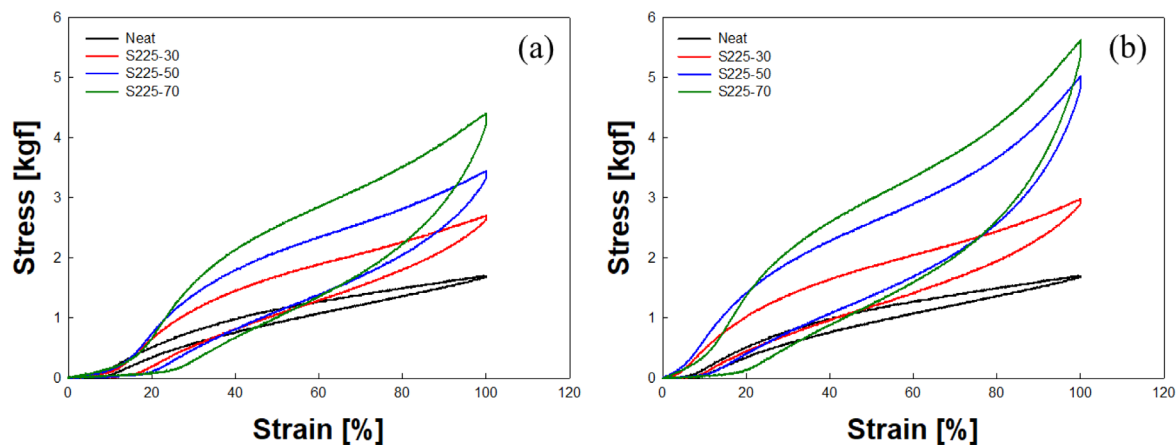


Figure 12. Hysteresis loss of EPDM/silica composite by silica content: (a) Before H₂ and (b) after H₂ exposure.

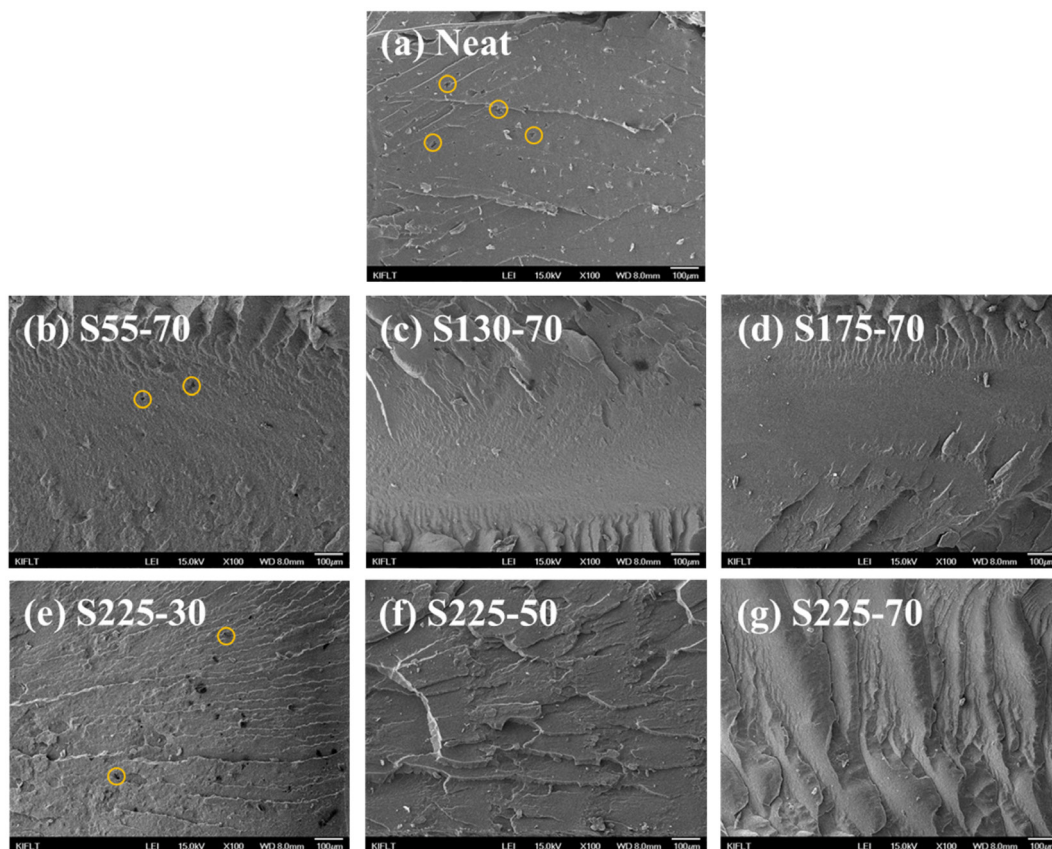


Figure 13. SEM images of the fractured surfaces of EPDM/silica composites after exposure to high-pressure hydrogen gas.

surface area silica at moderate loadings provides an optimal balance between reinforcement and resistance to hydrogen-induced degradation.

6. Image Analysis of EPDM/Silica Composites after High-Pressure Hydrogen Exposure

To analyze microscale defects in the EPDM/silica composite structure that may arise from exposure to high-pressure hydrogen, SEM images of the surface were obtained and are shown in Figure 13.

Defects attributed to hydrogen exposure were observed in the neat specimen (Silica 0 phr), along with in composites containing silica at low loadings and with relatively low specific surface area. In contrast, defect density decreased with increasing silica loading and with increasing silica specific surface area; no discernible defects were observed at high silica loadings. Notably, despite this reduction in hydrogen-induced voids, silica agglomeration became more severe with increasing silica content, suggesting a deterioration in dispersibility at elevated loadings.

Conclusions

This study demonstrated that the specific surface area and loading of silica play a decisive role in the hydrogen resistance and mechanical stability of EPDM/silica composites. High surface area silica enhanced crosslink density, reduced hydrogen permeability, and improved dimensional and mechanical stability by promoting strong polymer-filler interactions and restricting chain mobility.

Among the tested formulations, S225-50 exhibited the most balanced performance, combining low hydrogen permeability, superior tensile strength retention, minimal swelling, and stable viscoelastic behavior under high-pressure hydrogen exposure. These findings suggest that optimizing both the surface area and loading of silica provides a practical design strategy for rubber-based sealing materials in hydrogen storage and transport systems, where barrier efficiency and long-term durability are essential.

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Conflict of Interest: The authors declare that there is no conflict of interest.

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