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INFLUENCE OF m-ZnO ON THE THERMAL, MORPHOLOGICAL, AND CROSSLINKING BEHAVIOR OF EPDM UNDER GAMMA IRRADIATION

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ABSTRACT

Ethylene propylene diene monomer (EPDM) is a versatile elastomer known for its outstanding resistance to heat, oxygen, and radiation, making it suitable for applications involving prolonged exposure to ionizing radiation. This study evaluates the potential of zinc oxide microparticles (m-ZnO), traditionally used as activators in rubber compounding, to serve as effective radiation absorbers when incorporated into EPDM. Five formulations were developed with m-ZnO contents of 2, 4, 6, 8, and 10 parts per hundred rubber (phr) and exposed to gamma irradiation doses of 10, 100, and 1000 kGy. The results revealed that increasing m-ZnO content led to a rise in the glass transition temperature (Tg), indicating strong filler-polymer interaction and improved thermal stability. SEM confirmed uniform dispersion of fillers without aggregation, while irradiated samples showed intact morphology devoid of cracks or pores, highlighting the shielding role of m-ZnO. Gel fraction analysis further demonstrated that m-ZnO effectively reduced excessive crosslinking by absorbing gamma energy. However, at higher filler loadings, an opposite trend emerged, suggesting a saturation point beyond which the shielding efficiency diminished. Overall, the findings establish m-ZnO as a promising filler to enhance radiation resistance in EPDM composites, contributing to the development of sustainable and durable materials for nuclear, aerospace, and medical applications.

Keywords: EPDM, ZnO Microparticles, Gamma Irradiation, Radiation Shielding, Gel Fraction, Glass Transition, Morphology

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INTRODUCTION

Gamma radiation presents serious risks in the nuclear and medical sectors, demanding the use of protective materials that ensure human safety. Elastomers like EPDM rubber are widely employed as seals and insulators due to their flexibility, lightweight structure, and ease of fabrication. However, their susceptibility to radiation-induced degradation through chain scission and crosslinking can compromise structural integrity and performance over time.[1] Polymeric materials are susceptible to radiation compared to more radiation-resistant materials like metals, ceramics, or glasses, and they can degrade at relatively low doses. The radiation effects can lead to cross-linking or scission in the polymer chains, causing changes in the macroscopic properties such as embrittlement, reduced elasticity, hardening, and ultimately material failure.[2] Degradation can impact the elastomer's effectiveness in critical applications, potentially jeopardizing safety if not addressed. To mitigate these effects, researchers are focusing on enhancing elastomer formulations through the addition of radiation-resistant fillers, such as introducing aromatic rings, metal oxides, hetero and heavy atoms.[3] These radiation-resistant fillers can effectively absorb radiation and stabilize the polymer by dissipating absorbed energy, thereby minimizing structural damage.[4] Additionally, metal oxides act as reinforcing agents, helping to maintain the polymer's structural integrity.[5] Mahmood et al. reported the effects of various CaO loadings on EPDM and showed the enhanced mechanical, thermal, and electrical characteristics. Suntako examined m-ZnO in chloroprene (CR) foam, finding that nano-sized ZnO significantly enhanced crosslinking and mechanical strength. The high specific surface area of m-ZnO promotes better bonding and reduces the required amounts for performance similar to that of conventional ZnO.[7] Alsayed *et al.* explored ZnO/HDPE composites, comparing bulk and n-ZnO. The study discovered that m-ZnO improved effectiveness in gamma radiation shielding by increasing mass attenuation

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coefficients.[8] Lim-aroon *et al.* developed lead-free natural rubber (NR) sponges for X-ray and gamma-ray shielding, utilizing bismuth oxide (Bi_2O_3) as a filler and Oxybis (benzene sulfonyl) hydrazide (OBSH), an aromatic compound introduced as a blowing agent. Studies revealed that higher Bi_2O_3 content improves radiation attenuation.[9] Poltabtim *et al.* investigated gamma-ray shielding properties of EPDM rubber composites enhanced with metal oxides (Fe_3O_4, W_2O_3, and Bi_2O_3) as lead-free alternatives for radiation protection. Their findings indicated that increasing metal oxide content demonstrated a durable, flexible gamma shielding material.[10] Despite these advancements, limited studies have addressed the role of m-ZnO in EPDM under high gamma doses. This study investigates the impact of increasing m-ZnO content (2–10 phr) on the thermal, morphological, and gel fraction behavior of EPDM composites before and after exposure to 10, 100, and 1000 kGy. The findings aim to support the development of radiation-shielding elastomers for critical applications, aligning with SDG 12: Responsible Consumption and Production by promoting sustainable material use in high-radiation environments.

EXPERIMENTAL

Material and Methods

Flexible EPDM material was developed using various compounding ingredients listed in Table-1. EPDM (KELTAN 9650Q) was purchased from LANXESS India Pvt. Ltd. The compounding fillers, like carbon black used as a reinforcing agent, and IPPD (N-Isopropyl-N′-phenyl-1,4-phenylenediamine) used as an antioxidant, were purchased from Supple Rubber Chemicals Pvt. Ltd, New Delhi. Rubber processing oil (Gandhar Oil Refinery India Ltd., Mumbai) as a lubricant, TAIC 70 (triallyl isocyanurate) used as a cross-linking coagent, and DCP 40 (dicumyl peroxide) used as the vulcanizing agent, purchased from Lab Line Enterprises, Haryana. M-ZnO (< 5µm), stearic acid, and MAA (methacrylic acid) were purchased from Sigma-Aldrich chemicals Pvt. Ltd., Bangalore. The formulation details of mZnO/EPDM (mZE) composites are presented in Table-1

Table-1: Formulations of mZE Rubber

Material	Quantity	Function
Keltan 8570C	100 phr	Polymer EPDM
Carbon Black (N330)	50 phr	Reinforcing filler
Rubber processing oil 840	12 phr	Lubricants
Stearic acid	1.5 phr	Lubricants and Activator
IPPD (4010 NA)	3 phr	Antioxidant
TAIC 70	2 phr	Crosslinking coagent
MAA	15 phr	Crosslinking coagent
DCP 40	3phr	Vulcanizing agent
m-ZnO	2, 4, 6, 8, 10 phr	Activator and radiation absorber

EPDM is a terpolymer composed of ethylene, propylene, and a diene unit (ethylidene norbornene). The presence of the diene unit introduces unsaturation into the polymer chain, facilitating vulcanization or crosslinking. The ratio of each monomer in EPDM can be modified to optimize its heat and radiation resistance potential.[11] Ethylene contributes crystalline regions to the polymer, which are known to resist radiation more effectively than amorphous regions, resulting in enhanced stiffness, mechanical strength, and abrasion resistance. Propylene, on the other hand, reduces crystallinity, increases flexibility, and softens the polymer, making it suitable for applications such as hoses and seals that require elasticity.[12] The diene provides reactive double bonds that allow for the formation of crosslinks with agents such as sulphur or peroxides. However, a lower diene content is preferred in radiation-resistant applications, as excessive unsaturation can increase reactive sites for degradation under irradiation.[13] Keltan 8570C, containing 66% ethylene, 29% propylene, and 5% ENB, was used as the EPDM base. Carbon black, particularly N330, served as a reinforcing filler, improving mechanical strength and offering UV protection. It also helped dissipate heat during compounding.[14] Organic peroxide was chosen over sulphur for vulcanization due to its lower toxicity and greater resistance to gamma-induced degradation. Peroxide forms stable C–C crosslinks, unlike the less durable sulphur-based bonds.[15] Dicumyl peroxide (DCP) decomposes at high temperatures, enabling controlled curing and denser crosslinking networks.[16] DCP-40, with 40% active

DCP and 60% inert fillers, ensured safe handling and uniform mixing. In peroxide vulcanized systems, coagents like triallyl isocyanurate (TAIC) are incorporated to boost crosslinking efficiency. TAIC contains three highly reactive allyl groups that promote extensive crosslinking with both peroxide and polymer chains.[17] Methacrylic acid (MAA) reacts with ZnO to form zinc dimethacrylate, which results in an inorganic polymeric complex that increases crosslink density.[18] This enhances tensile strength, thermal stability, and tear resistance, and reduces compression set. Antioxidant IPPD is included to prevent oxidative degradation during the radical-driven peroxide curing process.[19] Rubber processing oil improves the workability, flow, and dispersion of fillers during mixing, while stearic acid acts as both a lubricant and a curing activator by promoting free radical generation for efficient crosslinking.[20,21]

General Procedure

m-ZnO Modification in EPDM

ZnO is generally included in the compounding of elastomers as it behaves as an activator and reinforcing agent. In peroxide vulcanized elastomer, ZnO serves as an activator for multifunctional co-agents that improve the crosslinking efficiency by reacting with polymer radicals generated by the peroxide and devising additional crosslinks.[22] Thus, the mechanical properties and thermal stability are enhanced. It also behaves as a chemical reinforcing agent, ameliorating the tensile strength, hardness, and abrasion resistance. ZnO can absorb and scatter radiation, hence playing a vital function in protecting against oxidation and UV degradation.[23] It can also defend against thermal degradation to some extent by neutralizing acidic by-products formed during curing. Traditionally, microparticles are used as they embrace a good symmetry in cost, ease of handling, and effectiveness in rubber formulations. With recent advancements and growing interest, microparticles are infused in EPDM elastomer at varying concentrations (2, 4, 6, 8, and 10 phr). m-ZnO can magnify the mechanical and thermal behavior of rubber more effectively than microparticles because of its elevated surface area, leading to enhanced reinforcement.[24] m-ZnO is more reactive, meaning less quantity will be required compared to conventional ZnO.

EPDM Compounding and Vulcanization

For compounding of mZE composite, all the ingredients listed in Table-1 are added and mixed using an internal Z blade kneader and two-roll mill for obtaining a homogenized mixture and to build a sheet-like structure. (Figure-1) displays the blending process using a two-roll mill and a photograph of various formulated sheets of mZE. A total of 5 batches of composite were blended with varying concentrations of m-ZnO (2, 4, 6, 8, and 10 phr) for about 20 minutes at 60ºC. After mixing all the components, the final step was the vulcanization and molding, where high temperature and pressure was required to initiate the crosslinking and molding into shapes, respectively. The required amount of elastomeric mixture from the roll mill was placed into a square-shaped mould with 2 mm thickness and 150 mm length, vulcanized at 160°C for 5 minutes under a 20 MPa pressure using a compression molding press to develop the final vulcanized elastomeric composites.[25]

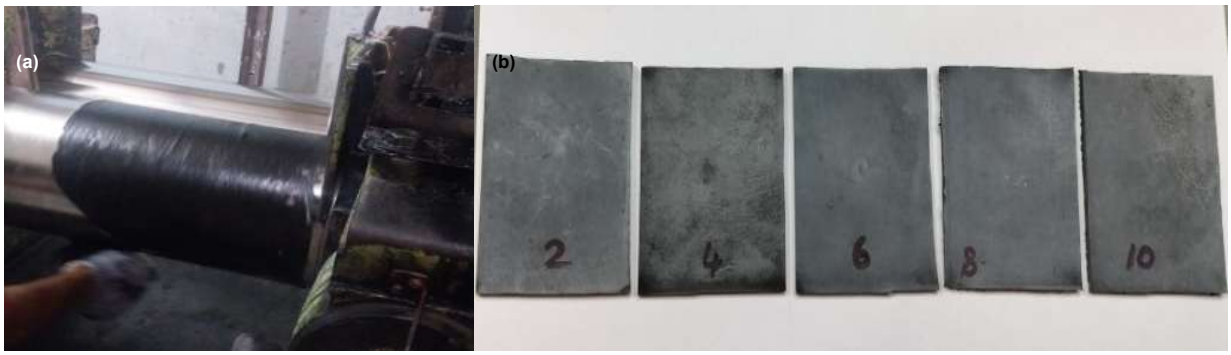


Fig.-1: (a) Blending of mZE using Two-Roll Mill, (b) Photograph of Final Cured mZE Composite Sheets

Gamma Radiation

Samples measuring 50x100 mm were extracted from the 2ZE, 4ZE, 6ZE, 8ZE, and 10ZE sheets and exposed to gamma rays in a gamma chamber equipped with Co-60 as the source. The samples were taken

out of the chamber at incremental dose intervals of 10, 100, and 1000 kGy in order to assess the radiation effects on their thermal, morphological, and crosslinking properties. (Figure-2) shows the photograph of samples irradiated at various dosages.

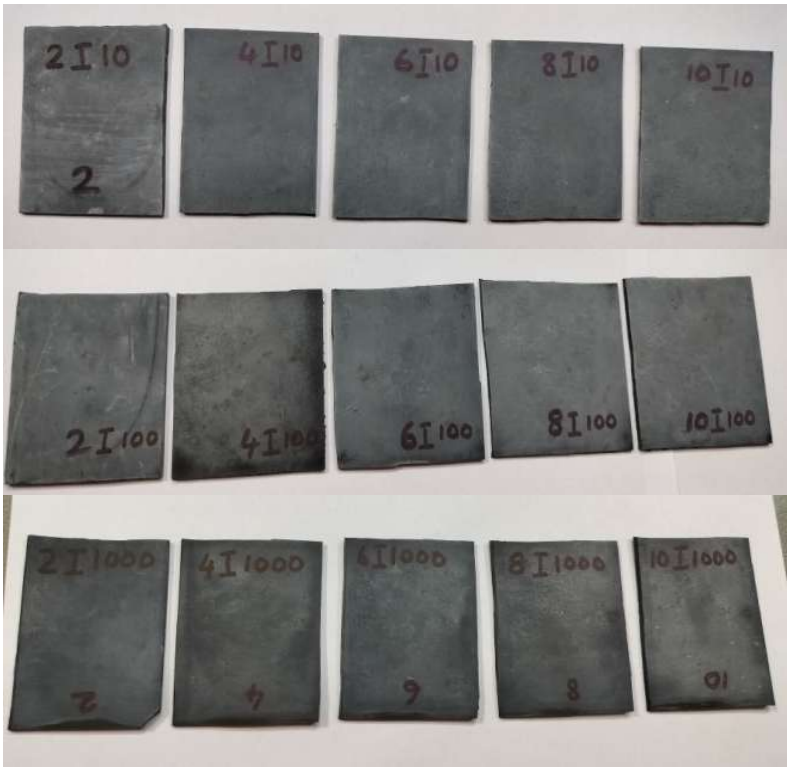


Fig.-2: Photograph of Sheets Irradiated at Various Dosages, 10, 100, and 1000 kGy

Detection Method

Thermal and Surface Morphology Test

Differential scanning calorimetric (DSC) testing was achieved using TA Instruments, USA, and Model Q20. For each run, a specimen weight of approximately 3-6 mg was utilized. Before the experimental runs, the instrument underwent calibration using pure indium standards. The thermal degradation of the range -80 to 450℃ was performed in the presence of commercial N_2 atmosphere with a temperature rate of 5℃/min.[26] The transitions obtained in the plot against heat flow (Mw/mg) and temperatures (℃) were used to determine Tg and other thermal events of both irradiated and non-irradiated samples. The morphology of mZE composites, both irradiated and unirradiated, was examined using the Vega 3 Tescan and Carel Zeiss EVO 18, with an accelerating voltage of 30 and 10 keV, respectively. As the composites were non-conducting, the surface of the elastomeric samples was overlaid with 5-10 nm of high-purity gold before the analysis.[27] The working distance between the sample and the SEM lens was maintained between 10 and 200 micrometers, with magnifications from 200x to 2000x.

Swelling and Gel Fraction Test

Elastomers such as EPDM, natural rubber, styrene-butadiene rubber, and butyl rubber generally exhibit high reactivity toward non-polar hydrocarbons and resistance to polar solvents like water, methanol, acetone, and glycerol.[28] This behaviour is primarily attributed to the molecular configuration of these elastomers. For example, EPDM consists mainly of ethylene and propylene units, which create long, saturated, non-polar hydrocarbon chains with minimal or no polar functional groups. According to the principle of "like dissolves like," non-polar solvents tend to interact more effectively with non-polar materials, whereas polar solvents exhibit limited interaction. As a result, non-polar solvents such as hexane, toluene, xylene, and cyclohexane are typically employed in analysing the swelling behaviour and gel fraction (indicative of crosslinking density) of elastomeric systems.[29] In the swelling test, elastomer samples are immersed in a chosen solvent and maintained at an elevated temperature for a defined period. During

this process, the solvent penetrates the polymer network and occupies the free volume or voids within the matrix, causing the sample to expand or swell. The extent of swelling is inversely related to the crosslink density of the elastomer; a highly crosslinked network restricts solvent penetration, resulting in lower swelling, while a loosely crosslinked or uncrosslinked network allows greater absorption and expansion.[30] (Figure-3) illustrates the absorption stages of solvent by elastomeric materials.

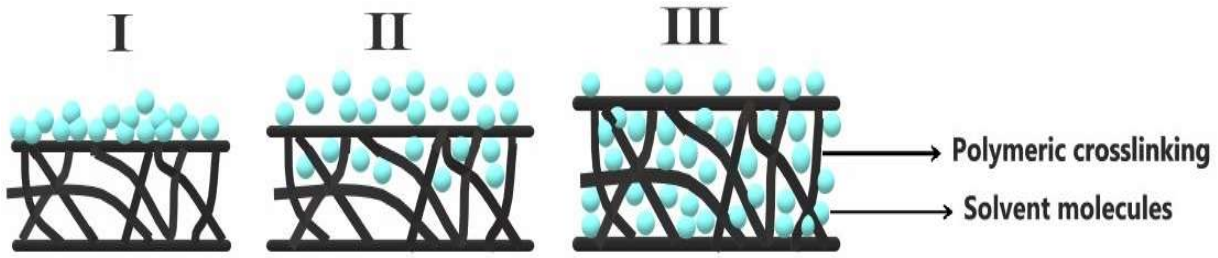


Fig.-3: Pictorial Representation of Polymeric Material under Swelling Mechanism

The swelling was reversible, unless after prolonged exposure. If the rubber was removed from the solvent and dried, it might return to its original proportions with some decline in weight due to the dissolution of broken polymer caused by chain scission. The ratio of the two weights shows the level of insoluble crosslinking called gel fraction. The outcome of increasing m-ZnO and radiation effects in EPDM could be illustrated from the gel fraction obtained. The gel content of the samples was assessed by boiling 20 mm x 20 mm samples in o-xylene for 24 hours in a closed vessel, following the American Society for Testing and Materials (ASTM) D2765 standards. (Figure-4) illustrates the reaction chamber at various time intervals and the swelled EPDM. Each formulation underwent this reaction three times, and the average results were used. Once the reaction was completed, the samples were dried in a hot air oven at 60 °C for 5 hours to remove the absorbed solvent. The weights before and after oil swelling was recorded, and the gel content (in %) was determined using the following equation 31:

$$\text{Gel Content (\%)} = \frac{\text{Weight after extraction } (W_f)}{\text{Weight before extraction } (W_i)} \text{ X } 100$$

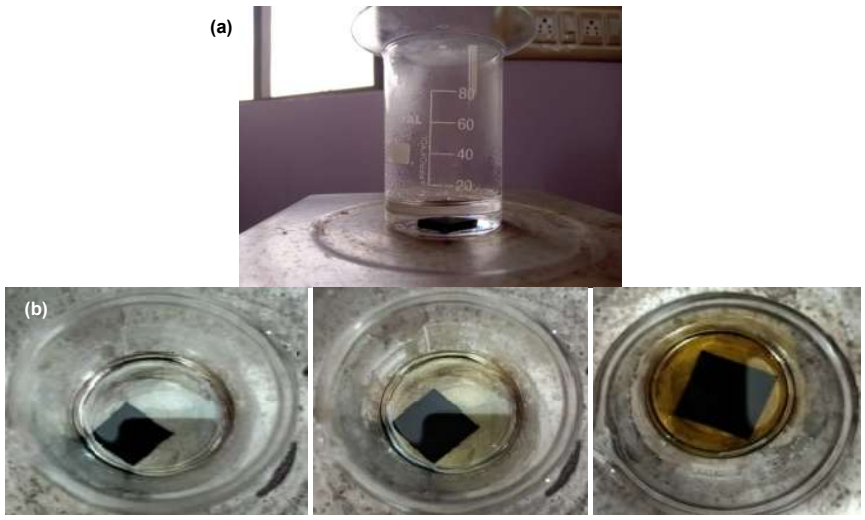


Fig.-4: Photograph Showing (a) Swelling of Specimen in Xylene, (b) Visual Representation of Specimen Expansion at Various Time Intervals (2, 10, and 20 hrs)

RESULTS AND DISCUSSION

Effect of Radiation on Elastomers

Gamma irradiation significantly affects elastomers by initiating chemical reactions such as crosslinking and chain scission. Crosslinking is a covalent bond linking two polymeric chains through a vulcanizing agent.

Under external conditions like prolonged exposure to UV light, high temperature, and gamma rays, induced crosslinking occurs. Some degrees of crosslinking can improve mechanical strength and rigidity, but excessive crosslinking can make the material brittle, with negligible elasticity, and lose functionality.[32] Chain scission is the breaking of polymeric material into smaller components due to factors such as radiation, heat, or oxidation. Protracted vulnerability to radiation in the presence of oxygen accelerates chain scission. As oxidation persists, the oxygen molecule reacts with free radicals induced by irradiation, forming peroxy radicals that abstract hydrogen atoms from polymeric chains, forming hydroperoxides, leaving behind a cleaved, shorter polymer chain. This leads to the fragmentation of chemical bonds within the polymer backbone, causing chain scission. The longer the exposure to irradiation at oxidative conditions happens, the more pronounced scission occurs, weakening the material by reducing its molecular weight, elasticity, and mechanical strength, ultimately leading to application failure.[33]

Thermal Analysis

The DSC curve for varying formulations of mZE before and after irradiation was shown in Fig.-5. The incorporation of m-ZnO resulted in a slight increase in the glass transition temperature (Tg), which can be attributed to enhanced filler–polymer interactions. This interaction suggests a restriction in polymer chain mobility, contributing to improved strength and thermal stability of the composite. In irradiated samples, a marginal yet consistent rise in Tg was also observed.

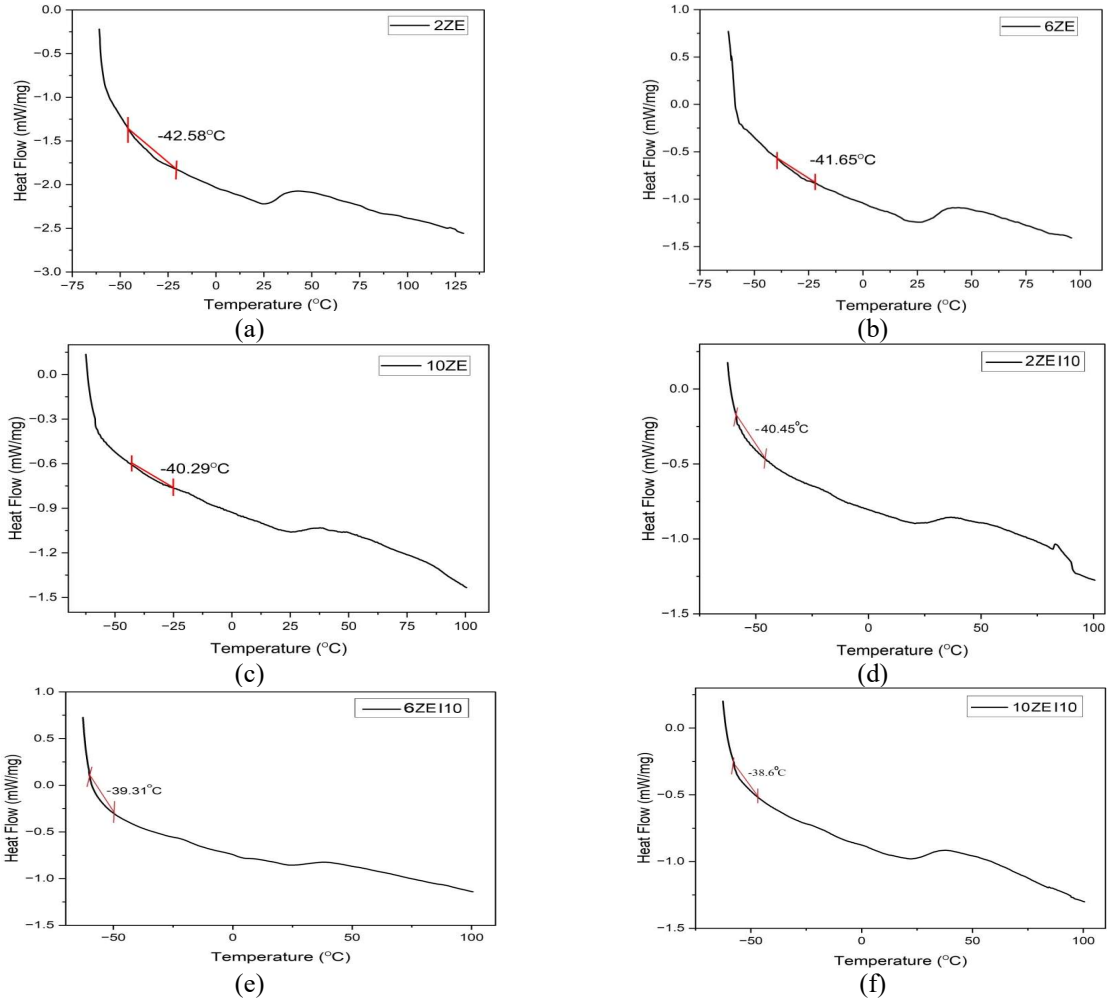


Fig.-5: DSC Thermograms (a–c) Before Irradiation of 2, 6, and 10 phr mZE, respectively, and (d–f) After Irradiation of 2, 6, and 10 phr mZE, respectively

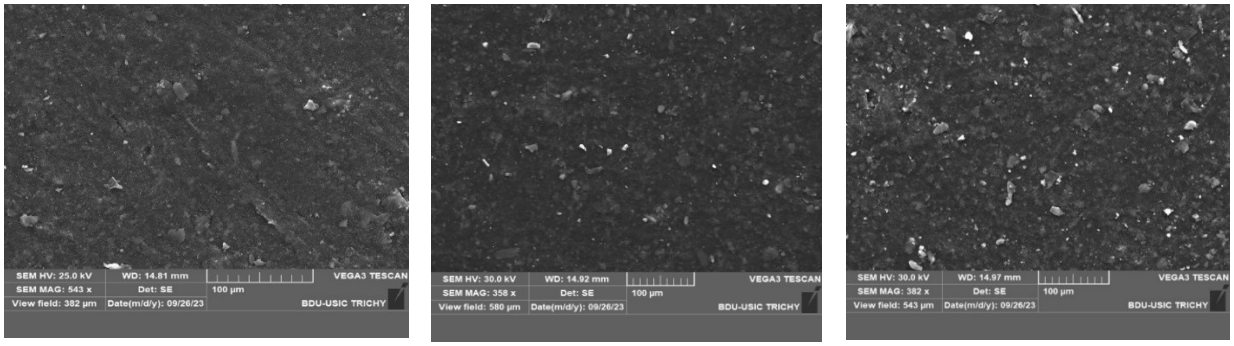
(b)

This increase is likely due to the ability of m-ZnO to dissipate the energy from incoming gamma rays, either through absorption or scattering mechanisms. By reducing the direct impact of radiation on the polymer

backbone, m-ZnO appears to play a protective role, thereby minimizing radiation-induced degradation. The suppression of chain scission and preservation of the network structure result in a more thermally stable material. This stabilization effect is reflected in the elevated Tg values of irradiated composites, supporting the conclusion that m-ZnO contributes positively to the radiation shielding and thermal endurance of EPDM-based materials.[34]

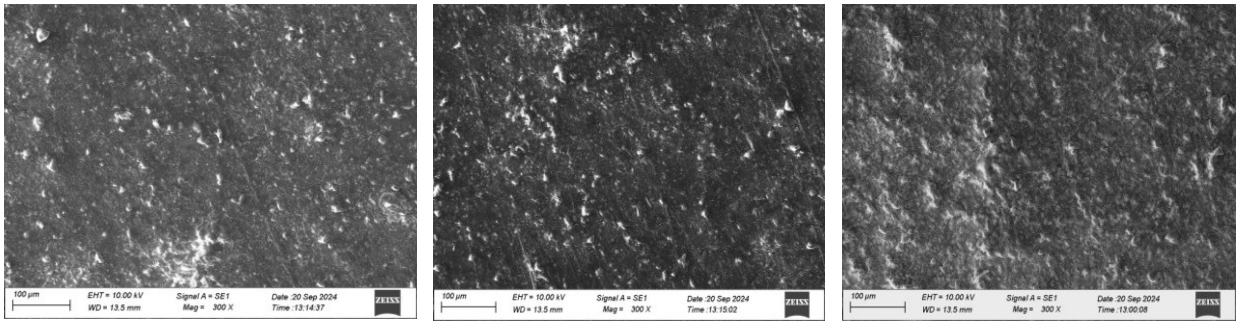
Morphological Studies

The SEM images of mZE composites before and after irradiation are shown in (Fig.-6) and (Fig.-7). SEM images in Fig.-6(a–c) illustrate the enhanced and consistent distribution of the m-ZnO filler without aggregates, which was due to the controlled processing conditions employed during sample manufacturing.[35] SEM image in Fig.-7(a–c) depicts the elastomeric surface of mZE composite at various dosages of gamma irradiation. Even after gamma irradiation, the SEM images reveal a smooth, intact morphology with no evidence of porosity or crack formation, likely due to the ability of m-ZnO to enhance crosslink density and reinforce the rubber matrix for improved structural integrity and stress distribution.



(a) (b) (c)

Fig.-6: SEM image of (a) 2ZE, (b) 6ZE, (c) 10ZE Composite Before Irradiation



(a) (b) (c)

Fig.-7: SEM image of (a) 2ZE, (b) 6ZE, (c) 10ZE Composite after Irradiation

Gel Fraction Determination

Crosslinking was a crucial process in rubber manufacturing, furnishing essential strength and resilience. However, during service in demanding applications such as tires, pressure seals, and other applications where there is a necessity for exposure to atmospheric oxygen, heat, and high radiation can induce excessive crosslinking. This makes the material more rigid, brittle, and prone to cracking under stress. The heat tolerance gets diminished, which results in premature degradation. The gel fraction mechanism involves the selective interaction of solvent with the polymer matrix: the crosslinked network remains unaffected by the solvent, while smaller chains or those with scissioned bonds dissolve into the solvent. The influence of varying concentrations of m-ZnO and different doses of gamma radiation on crosslinking was investigated, focusing on how each factor impacts the crosslinking density within the elastomeric structure. The Gel fraction for all formulated mZE sheets was determined using the gel fraction equation and plotted as a function of radiation (Fig.-8) and filler dosage (Fig.-9). Figure-8 shows that the gel fraction (%) increases with increases in radiation dosage. With increasing gamma radiation in the presence of oxygen, both crosslinking and chain scission occur concurrently. Initially, free radicals promote stable crosslink

formation, increasing crosslink density. At higher doses, although chain scission intensifies, the results show continued crosslinking, likely due to transient entanglements from scission that do not hinder permanent network formation.[36] Thus, crosslinking dominates, enhancing structural integrity under irradiation. In the m-ZnO composite, m-ZnO originally lowers the crosslinking density, up to 6 phr of m-ZnO, and a further increase in the concentration of m-ZnO shows an accumulation in the crosslinking density. The m-ZnO particles could absorb the radiation and hamper the formation of crosslinking and entanglement coupling. However, at a higher dosage of m-ZnO filler, its tendency to block the crosslinking was downsized. On excess dosage, an unexpected effect occurs, secondary radiation effect, and enhanced free radical formation. Since m-ZnO absorbs high-energy radiation, it could emit secondary radiation and facilitate free radical formation, resulting in increased crosslinking.

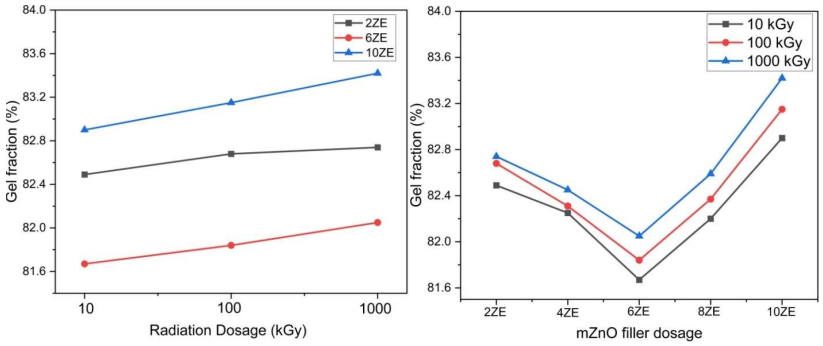


Fig.-8: Gel Fraction of mZE Composites as a Function of (a) Radiation Dose, (b) m-ZnO Filler Dosage

CONCLUSION

The investigation underscores the role of m-ZnO in enhancing the performance of EPDM rubber under gamma irradiation. M-ZnO improved thermal stability and crosslink density by effectively dissipating gamma rays, as demonstrated by the increase in Tg with higher m-ZnO concentrations. SEM analysis confirmed consistent dispersion and structural integrity, even at elevated radiation doses. Swelling tests reflected reduced polymer degradation and enhanced resistance to solvent penetration, correlating with decreased crosslinking formation, preventing the risk of brittleness and cracking, and increasing the lifespan of the utility. This work marks the first study highlighting the m-ZnO as a sustainable and effective additive for developing radiation-resistant elastomeric materials. This research aligns with SDG 12 – Responsible Consumption and Production by promoting sustainable material development to mitigate gamma-ray–induced damage.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-12: Responsible Consumption and Production*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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