Peroxide-Cured Silicone Rubber Composites: Structural, Morphological, and X-ray Detector Performance Analysis

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Abstract

In this study, a soft and flexible polymer composite of silicone rubber was synthesized by incorporating varying concentrations of a peroxide curing agent using the solution casting method, a cost-effective method at room temperature. The synthesized composites were characterized through Energy Dispersive X-ray Analysis (EDAX) and Scanning Electron Microscopy (SEM) to determine elemental composition and surface morphology. SEM micrographs revealed a smooth surface with uniformly distributed crystallites, forming a matrix-like arrangement. Additionally, X-ray switching response measurements were recorded at different voltages to evaluate the detector performance of the composite material. It was observed that detector is showing the photo-response but the difference in photocurrent and dark current needs to be enhanced by adding conducting fillers in the matrix of silicone rubber. The results suggest that peroxide concentration significantly influences the structural, morphological, and detector response properties of the silicone rubber composite, offering valuable insights into optimizing curing conditions for enhanced material performance and detector efficiency.

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Introduction

In this paper we use peroxide cured Silicon rubber. Silicone rubber is a highly versatile elastomer widely recognized for its exceptional thermal stability, chemical resistance, and flexibility, which makes it indispensable across various industries, including medical devices, automotive components, and electronics.

The polymer class known as silicone rubber has special qualities like electrical insulation, biocompatibility, and tolerance to both high and low temperatures. Numerous industries, such as the electrical, medical, and aviation sectors, heavily rely on silicone rubber [1]. Three primary methods are used to create silicone rubber: condensation vulcanization, peroxide curing, and platinum-catalyzed curing.

According to published research, the earliest technique for vulcanizing silicone rubber was peroxide curing, often known as radical curing. Since peroxides start the radical crosslinking reaction, it can be challenging to get rid of some of the tiny molecules created during this process from the synthetic silicone rubber. These tiny molecules can occasionally degrade silicone rubber's mechanical qualities and usage [2]

Silicone rubber composites have garnered significant attention due to their exceptional flexibility, chemical stability, and adaptability across various applications. Poly(siloxane)s of high molecular weight, known as silicone rubbers, are materials based on macromolecules with successive silicon–oxygen bonds as a backbone, with the Si atom having two monovalent organic side groups [3]. Polysiloxanes are materials composed of a (Si–O) backbone with two monovalent organic radicals attached to each silicon atom (–R_2Si–O–). They are commonly referred to as "silicone" polymers; Kipping coined this term in 1901 to characterize novel compounds with the formula R_2SiO by drawing a comparison to ketones. Other names for these kinds of polymers are siloxane polymers and polysiloxanes since the (–Si–O–) repeat unit is also known as the "siloxane" bond or linkage. Silicones serve as a crucial link between inorganic and organic polymers since their backbone is "inorganic" and the substituents that are joined to the silicon atom are typically "organic" radicals [4].

These materials are particularly promising for radiation detection, leveraging their intrinsic properties to act as effective X-ray detectors [5]. The base composition of silicone rubber, represented as Silicone rubber composites have garnered significant attention in recent years due to their remarkable flexibility, chemical stability, and adaptability across diverse applications. These materials are particularly promising for radiation detection, leveraging their intrinsic properties to act as effective X-ray detectors. This study investigates peroxide-cured silicone rubber as a soft, flexible material for X-ray detection applications. Digital radiography, dental X-ray imaging, CT scans, and radiation therapy are just a few of the medical diagnostic applications that use low-energy, pulsed X-rays. Even with commercial X-ray detectors in use today, it can be difficult to get consistent, high detector performance over time due to X-ray interactions with the detector material. Researchers must envision new generation X-ray detector materials that are lightweight, portable, economically feasible, simple to produce, and have mechanical flexibility and low-temperature capabilities and high temperature. The discovery of X-rays is regarded as a major scientific breakthrough with numerous uses in the fields of business, medicine, astronomy, and warfare [6]. Certain detectors with great sensitivity, cheap fabrication costs, and high stability at room temperature are necessary for explicit applications [7]. However, these rays have some major negative health impacts in addition to being useful in a variety of applications. Wearable sensors that can function at room temperature with low biased voltage and have good sensitivity to X-rays are necessary to regulate any occupational and war exposures. By employing a cost-effective and laboratory-friendly preparation method that avoids any temperature and mechanical stresses, structurally stable and defect-free materials were synthesized.

Characterization using SEM revealed uniform surface morphology and well-aligned granules, confirming the material's structural integrity. Additionally, EDX analysis indicated the presence of key elemental components such as carbon, oxygen, copper, and hydrogen.

Preliminary X-ray sensing experiments demonstrated photo response behaviour in the material, though with limited differentiation between photocurrent and dark current. To address this, the incorporation of fillers is proposed to enhance the photo response and optimize the material's performance as an X-ray detector. For Further characterization, including XRD analysis, is planned to gain deeper insights into the structural and functional properties of the composite. This work lays the foundation for developing cost-effective, high-performance silicone rubber composites for advanced radiation detection systems.

Method

Experimental Methodology

Sample Preparation

To investigate the effects of curing agent concentration on the properties of silicone rubber, samples were prepared using the solvent casting method [8] at room temperature. This method is advantageous for producing uniform thin films composites for X-ray switching applications at room temperature. Films of varying compositions are synthesised by mixing five ml of silicone rubber with varying amounts of curing agent as tubulated in Table 1. The silicone rubber and curing agent were dissolved in an appropriate solvent to ensure complete homogenization. The solutions were then poured into mould and left to dry at room temperature. The solvent evaporation process took approximately one week, allowing for the formation of well-structured composite films. This approach aligns with methodologies used in the preparation of polymer composites for high-performance, room-temperature direct X-ray detectors [5].

Table 1: Samples of Peroxide – Cured Silicon rubber with varying amounts of curing agent.

Sample	Amount of Silicone Rubber (ml)	Amount of Curing agent (ml)
Sc 1	5	1
Sc 2	5	2
Sc 3	5	3

Curing Process

After solvent evaporation, the samples underwent a curing process at room temperature. Room temperature curing is beneficial for maintaining the integrity of certain additives and preventing thermal degradation, which is crucial in the development of sensitive detection materials. The curing agent initiates a free-radical mechanism, promoting polymer cross-linking through peroxide-induced reactions. This process contributes to the material's flexibility and mechanical properties, essential for potential use in X-ray detectors.

Physical Characterization

Following preparation, the physical properties of the samples were characterized by measuring thickness, density, diameter, and mass. These parameters are critical as they directly influence the mechanical and functional performance of silicone rubber composites, particularly in sensor applications like X-ray detectors.

Thickness: Measured using a micrometer to ensure uniformity across samples. Uniform thickness is vital for consistent detector response, as variations can affect the material's sensitivity to X-rays.

Density: Calculated by measuring the mass and volume of each film. Density variations can indicate differences in cross-linking density, affecting the material's electrical properties, which are crucial for detector performance.

Diameter and Mass: Measured using a calliper and an analytical balance, respectively. These measurements help correlate the effects of curing agent concentration on the overall performance of the material, as mass and dimensional stability are important for the integration of detector materials into devices.

These physical characteristics of three samples are tabulated in Table 2.

Table 2: Physical characteristics of Peroxide-Cured Silicon rubber samples.

Sample	Thickness (cms)	Mass (gms)	Diameter (cms)	Density (gm/cm^3)
Sc 1	0.38	6.49	4.5	1.074
Sc 2	0.45	7.48	4.5	1.045
Sc 3	0.41	6.82	4.5	1.045

The results from these physical characterizations are essential for correlating the mechanical and functional properties of the samples to their X-ray detection capabilities, which will be evaluated in subsequent sections.

Results and Discussion

Physical Structural Analysis

The solvent casting method yielded uniform composite films with varying thicknesses and densities based on curing agent concentration. Higher concentrations resulted in denser cross-links, improving mechanical strength but slightly reducing flexibility, consistent with findings from previous studies [6].

SEM Investigations

The SEM analysis shows that the synthesized silicon rubber-based material exhibits a uniform surface morphology. Uniformity in the surface indicates consistency in the material preparation process and reflects the absence of unwanted aggregation or segregation of components. The SEM images (Figure 1(a) and 1 (b)) under different magnifications highlight aligned granules across the material's surface. Granular alignment suggests that the internal structure is well-organized, which could contribute to efficient charge transport when exposed to X-rays [7]. Interconnection among aligned granules could provide necessary pathways for charge carriers during charge transport mechanism.

The SEM did not reveal major deformities, such as cracks, voids, or irregularities. These defects typically arise from high-temperature or high-pressure processes [8], which were avoided in this material's synthesis due to the cost-effective, low-stress preparation technique.

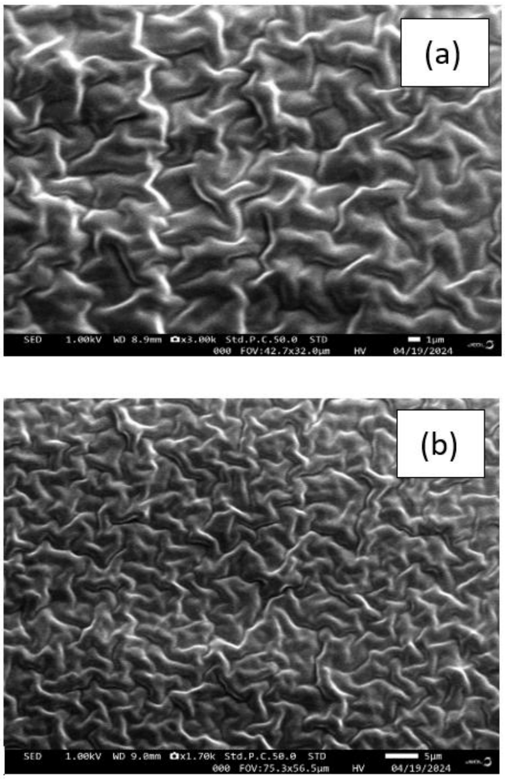

Figure 1: (a) and **(b)** depicting aligned granules peroxide cured Silicone Rubber composite films at two magnifications namely 2 and 5 micrometres.

The absence of cracks or deformities ensures that the material maintains its mechanical and structural integrity over time and thus provide stability to the structure. A defect-free material promotes uniform electric field distribution and minimizes the scattering of charge carriers, improving sensitivity and reliability and thus helps in achieving better charge transport characteristics.

It is expected that with smooth and periodically aligned granules at microlevel, the composite films are less likely to degrade under repeated exposure to radiation or stress, enhancing its lifespan as a detector.

Energy Dispersive X-ray Analysis

The Energy-Dispersive X-ray Spectroscopy (EDX) analysis provides valuable insights into the elemental composition of the silicon rubber-based material.

(a)

(b)

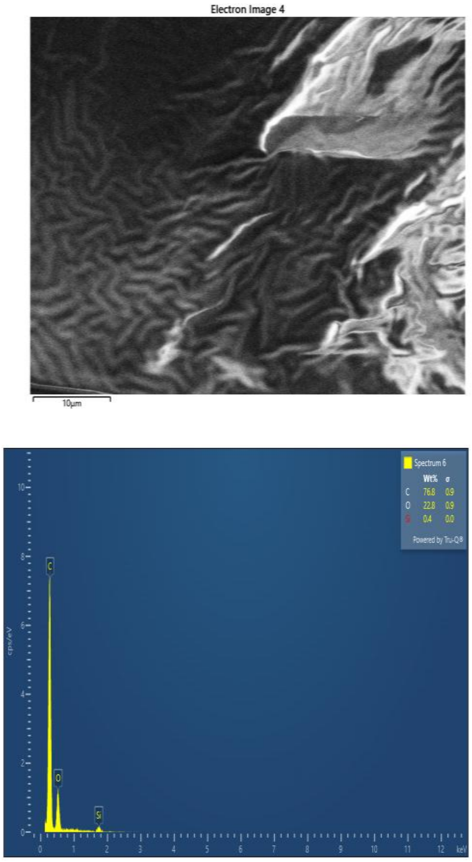

Figure 2: EDAX of sample SC-1

(a)

(b)

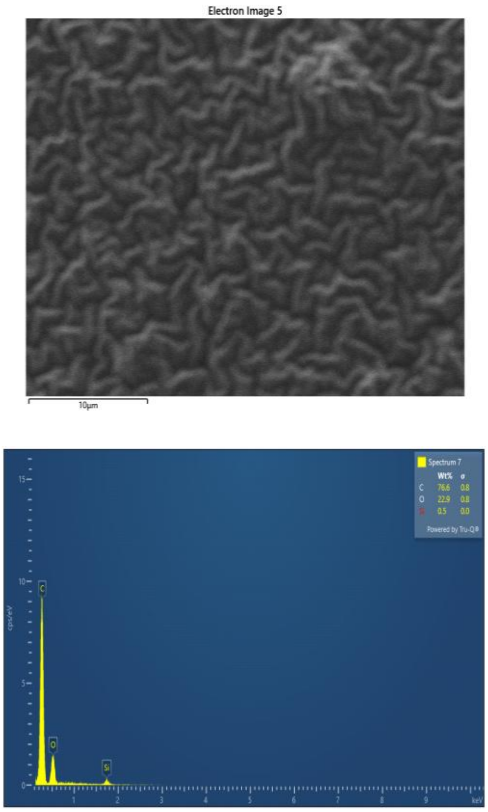

Figure 3: EDAX of sample SC-2

(a)

(b)

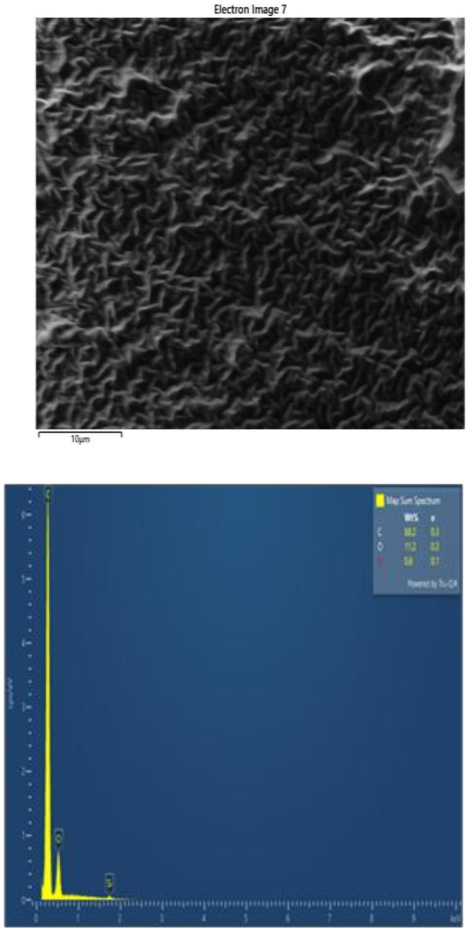

Figure 4: EDAX of Sample SC-3 confirms the homogenous mixture of the materials.

The EDAX analysis showed presence of Silicon, Oxygen and Carbon peaks corroborating with materials used for synthesising the composite films.

The presence of various elements present as per EDAX analysis is due to following factors. Carbon Predominantly originates from the organic groups (RRR) in the silicon rubber backbone, $(R_2SiO)_n(R_2SiO)_n(R_2SiO)_n$. Carbon contributes to the material's flexibility and soft texture, which are essential for mechanical stability and ease of fabrication. Silicon is present as part of the silicon rubber base material. It plays a crucial role in enabling the material to interact with X-rays, as silicon atoms can absorb and scatter X-ray photons effectively. The Presence of Oxygen is linked to the SiO-SiO-SiO structure in the material’s framework. The presence of oxygen is critical for maintaining chemical stability and forming strong bonds in the polymeric structure. Hydrogen is implicitly present as part of the organic groups in the rubber contributes to the flexibility of the material, although its exact detection may be limited due to EDX's elemental detection range. Thus, the combination of silicon, oxygen, and carbon provides a robust base for detecting X-ray radiation. Silicon and oxygen enhance the material's ability to interact with X-rays, while carbon ensures the mechanical flexibility of the detector. The elemental composition indicates potential for doping or functionalization. Adding elements like copper could improve electrical conductivity. Incorporating additional elements or fillers might enhance photo-response sensitivity. The balanced presence of silicon, oxygen, and carbon ensures the material's resistance to degradation under X-ray exposure or environmental factors like humidity and temperature. Thus, these elements may provide chemical stability to the compound under Xray irradiations. The flexibility of the $(R_2SiO)_n(R_2SiO)_n(R_2SiO)_n$ framework allows for further modifications, such as integrating nanoparticles or other dopants to tailor the material's properties.

Photo-response Analysis

The photocurrent measurements for composite samples irradiated with 30 kV X-rays at a bias voltage of 210 V reveal significant insights into the impact of curing agent concentration on detector performance. For samples Sc 1 and Sc 2, the photopeak current was comparable to the dark current, indicating suboptimal sensitivity likely due to limited charge carrier mobility or high trap density. In contrast, sample Sc 3, which had a higher concentration of the curing agent, exhibited significantly better performance. This improvement is attributed to the robust matrix formed by the combination of silicon, oxygen, and carbon, which provided an effective framework for detecting X-ray radiation. The optimized 1:3 ratio of silicone rubber to curing agent created interconnected channels for charge carriers generated during X-ray irradiation, enhancing mobility and reducing trap density. However, the findings also emphasize that achieving an optimal balance between cross-link density and flexibility is critical to optimizing the performance of such detectors. A higher curing agent concentration improved charge carrier pathways and sensitivity, but excessive rigidity must be avoided to maintain mechanical integrity and functionality.

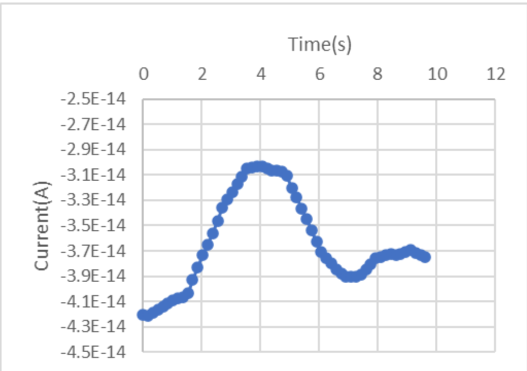

Figure 5: Depict the photocurrent response to the X ray-irradiation for sample Sc 3.

Optimization Strategies for Photo-Response Efficiency

Several optimization techniques can be taken into consideration in order to further improve the peroxide-cured silicone rubber composites' photo-response efficiency. Among these is adjusting the peroxide concentration to strike the ideal balance between charge transport channels and cross-link density. Furthermore, adding dopants or sensitizing compounds could greatly enhance the production of charge carriers under X-ray radiation. Additionally, altering the electrode and composite interfaces' surfaces may increase carrier collection efficiency and lower recombination losses. These strategies will be the main focus of future research to improve the composites' sensitivity and stability even more.

Comparison with Other X-ray Detector Materials

Compared to conventional X-ray detector materials such as amorphous selenium (a-Se), lead halide perovskites, and organic semiconductors, the peroxide-cured silicone rubber composites offer unique advantages. While a-Se and perovskites typically exhibit higher intrinsic sensitivities, they are often limited by brittleness, toxicity, or environmental instability. In contrast, the silicone-based composites present superior mechanical flexibility, chemical resistance, and biocompatibility. These attributes make them particularly suitable for emerging flexible and wearable X-ray detection applications, despite some trade-offs in terms of charge carrier mobility and photo response intensity.

Discussion on Photocurrent and Dark Current Differentiation

The relatively low differentiation between photocurrent and dark current observed in the composites highlights a critical challenge in improving device performance. To address this, the integration of conductive fillers such as carbon nanotubes, silver nanowires, or reduced graphene oxide is proposed. These fillers can create percolative networks within the matrix, enhancing charge transport and reducing leakage currents. Additionally, exploring alternative curing methods like UV-induced curing or platinum-catalyzed hydrosilylation may allow better control over the composite's microstructure, minimizing defects that contribute to dark current generation.

Conclusion and Future Prospectives

This study highlights the potential of peroxide-cured silicone rubber composites for high-performance X-ray detectors. By varying the curing agent concentration, significant improvements in structural and detector properties were achieved. Future work will focus on integrating conductive fillers and exploring hybrid curing methods to further enhance detector efficiency and flexibility.

Photo Current refers to the electric current generated when the material is exposed to X-ray radiation. Dark Current refers to the residual electric current flowing through the detector in the absence of X-ray exposure. The difference between photo current and dark current is small. This implies that the material's ability to distinctly respond to X-ray exposure (signal-to-noise ratio) is limited. This low differential may limit the detector's efficiency and sensitivity. It may be concluded that this material needs to be explored further with addition of fillers or dopants to amplify the photo current without increasing the dark current. Adding fillers with higher electrical conductivity or X-ray sensitivity can enhance the photo current. Suitable candidates include metallic nanoparticles, conductive polymers, or dopants tailored to improve charge carrier mobility. Also, the electrode interface or measurement setup needs to be optimized as it could introduce noise or other inaccuracies, leading to a less significant distinction between photo and dark current. Developing better interfaces between the silicon rubber and electrodes could reduce dark current noise and improve the signal-to-noise ratio.

Impact of Fillers on Composite Properties

Fillers play a crucial role in defining the electrical and photoconductive properties of polymer composites. Factors such as filler type, particle size, surface functionalization, and dispersion quality significantly affect the resulting performance. For instance, nanostructured fillers can enhance the carrier mobility by reducing trap states and facilitating efficient charge percolation. In this study, future investigations will explore various filler systems to optimize the trade-off between mechanical flexibility, electrical conductivity, and X-ray sensitivity. A better understanding of filler-matrix interactions could lead to composites with improved photocurrent generation and reduced dark current leakage. We have tried to synthesize samples with Bismuth Triiodide as fillers but the studies are under process.

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