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Preliminary Characterization of Natural Rubber Sheets Formulated with Basic Bismuth Nitrate and PEG4000: Density and SEM-EDX Analysis

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Abstract - This study presents a preliminary characterization of natural rubber sheets formulated with basic bismuth nitrate and polyethylene glycol 4000. The sheets were prepared using ammonia-preserved field latex at six formulation levels: 0%, 3%, 5%, 7%, 9%, and 11%. A dry rubber content of 35%, selected from published reports, was used to estimate the dry rubber mass; the actual dry rubber content of the latex batch was not measured. Density was calculated by dividing the average final mass of each sheet by its final geometric volume. The measured densities at the six formulation levels were 0.832, 0.595, 0.624, 0.642, 0.795, and 0.824 g/cm³, respectively. The lowest density occurred at 3%, and density did not increase consistently as the formulation level increased. Surface morphology was examined by scanning electron microscopy in the control and 11% sheets. Energy-dispersive X-ray spectroscopy was used to determine relative elemental composition in selected surface regions. The measured bismuth mass percentages at the 3%, 5%, 7%, 9%, and 11% levels were 1.00%, 0.63%, 1.86%, 2.99%, and 0.32%, respectively. No quantitative bismuth value was reported for the control sample. These findings indicate variation in density, surface morphology, and local elemental composition. However, the results do not establish uniform bismuth distribution throughout the sheets or radiation attenuation performance. Further work should measure the actual dry rubber content, assess elemental distribution over multiple regions, and directly evaluate radiation attenuation.

Keywords: field latex, dry rubber content, surface morphology, elemental composition, formulation level.

I. INTRODUCTION

The application of X-rays in medical imaging, particularly in computed tomography (CT), plays an important role in supporting diagnosis by providing visualization of internal anatomical structures. Nevertheless, the use of ionizing radiation requires appropriate radiation protection measures to maximize the clinical benefits of examinations while limiting unnecessary exposure. The International Commission on Radiological Protection (ICRP) establishes justification and optimization as fundamental principles of radiation protection, emphasizing the need to balance clinical benefits with the control of radiation exposure [1]. In this context, the development of radiation shielding materials with physical characteristics suitable for radiological applications remains an important area of research.

Lead-based materials have long been used for radiation shielding because of their ability to attenuate ionizing radiation. However, concerns regarding lead toxicity and the need for more flexible protective materials have encouraged the development of alternative polymer-based composites. These materials combine a polymer matrix with a radiation-interacting

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filler, allowing their physical and mechanical properties to be tailored through the selection of constituent materials and their proportions [2]. Bismuth-containing materials have been investigated as potential lead-free shielding fillers because of the high atomic number of bismuth and its interaction with X-ray photons. Toyen et al. investigated natural rubber-bismuth oxide composites, considering both X-ray shielding performance and mechanical properties [3]. Xu et al. studied natural rubber-based composites containing bismuth for gamma-ray shielding applications [4]. These studies provide a scientific basis for further research on natural rubber composites containing bismuth-based fillers. However, their findings cannot be directly extrapolated to the natural rubber sheets formulated with basic bismuth nitrate in the present study because the filler chemistry differs and the present work focuses on density and SEM-EDX characterization rather than direct radiation attenuation testing.

Natural rubber (NR) is a suitable polymer matrix for preparing flexible sheets. In composite formulations, the properties of the resulting material depend on the filler type, matrix composition, and fabrication conditions. Therefore, characterizing the fabricated material is necessary before drawing conclusions about its physical properties or potential applications. For CT applications, the suitability of shielding materials also requires consideration of their potential effects on image quality and automatic exposure control. The American Association of Physicists in Medicine (AAPM) has noted that bismuth-based shielding in CT may affect image quality and produce variable exposure outcomes under certain conditions, particularly when automatic exposure control or tube current modulation is used [5]. These considerations provide context for further research on bismuth-containing materials, but their effects were not evaluated in the present study. Accordingly, the suitability of the prepared natural rubber sheets for CT shielding cannot be established solely from their density and elemental composition.

Density is a useful physical parameter for the preliminary characterization of composite materials and is calculated as the ratio of mass to volume. Although density describes a physical property of a fabricated sample, it does not directly establish its radiation attenuation performance. Zen et al. characterized silicone rubber-tungsten (SR-W) composites using density measurements and energy-dispersive X-ray (EDX) analysis and reported that density did not increase consistently across different compositions [6]. This finding highlights the importance of interpreting density based on the measured mass and dimensions of each sample rather than assuming a consistent relationship between filler content and density. The dry rubber content of the raw material must also be considered because fresh natural rubber field latex and concentrated latex can have different dry rubber contents [7]. In addition to density measurements, scanning electron microscopy (SEM) combined with EDX provides information on surface morphology and elemental composition in the analyzed regions [8]. However, EDX quantification describes the relative elemental composition of the analyzed region and does not, by itself, establish the mass fraction of a specific chemical compound throughout the entire material.

Based on these considerations, this study aimed to provide a preliminary characterization of natural rubber sheets formulated with basic bismuth nitrate ($Bi_5O(OH)_9(NO_3)_4$) and PEG4000 at formulation levels of 0%, 3%, 5%, 7%, 9%, and 11%. The characterization comprised density measurements and SEM-EDX analysis. Density measurements were used to compare the physical characteristics of the dried sheets based on their measured mass and geometric volume, while SEM-EDX was used to examine surface morphology and determine the relative elemental composition of selected surface regions. Because the actual dry rubber content of the field latex was not experimentally determined and EDX measurements were limited to selected regions, this study provides preliminary information on the fabricated sheets without establishing the uniformity of filler distribution or their radiation attenuation performance.

II. Experimental Procedure

1. Preparation of Natural Rubber Sheets

Natural rubber sheets were prepared using natural rubber field latex obtained directly from a local rubber farmer and preserved with ammonia. The filler was the commercial product Bismuth(III) nitrate alkaline for analysis (Supelco/Merck, product no. 1.01878; CAS No. 1304-85-4), whose label specifies the chemical formula $Bi_5O(OH)_9(NO_3)_4$. This material is referred to as basic bismuth nitrate throughout this paper. PEG4000 and distilled water were used as additional formulation components. Six basic bismuth nitrate formulation levels were prepared: 0%, 3%, 5%, 7%, 9%, and 11%. The 0% formulation

served as the control, without the addition of basic bismuth nitrate.

For each formulation, the initial mass of liquid latex (m_0) was recorded before calculating the mass of basic bismuth nitrate to be added. A dry rubber content (DRC) value of 35% was used as a calculation parameter to estimate the dry rubber mass in the initial field latex. This value was selected based on literature reports indicating that field latex generally contains approximately 25-35% dry rubber [7,9]. However, the actual DRC of the latex batch used in this study was not experimentally determined. Therefore, the value of 35% represents a literature-based calculation parameter rather than a measured property of the raw latex. ISO 7681:2024 specifies a method for determining DRC in natural rubber field latex [10], but this method was not applied in the present study. ISO 2004:2024 specifies requirements for concentrated natural rubber latex [11] and was not used to establish the DRC of the field latex examined here.

The mass of basic bismuth nitrate (m_{BBN}) was calculated using Equation (1):

$$m_{\mathrm{BBN}} = \frac{C}{100} \times 0.35 \times m_0 \quad (1)$$

where m_{BBN} is the mass of basic bismuth nitrate added (g), C is the selected formulation level (%), and m_0 is the initial mass of liquid latex before subtracting the formulation components (g). The estimated dry rubber mass was calculated as 0.35 times m_0.

The mass of liquid latex used for mixing (m_{latex}) was calculated by subtracting the masses of basic bismuth nitrate, PEG4000, and distilled water from the initial latex mass, as expressed in Equation (2):

$$m_{latex} = m_0 - m_{\mathrm{BBN}} - m_{PEG} - m_{aq} \quad (2)$$

The formulation mass balance was established by subtracting the masses of basic bismuth nitrate, PEG4000, and distilled water from the initial latex mass to determine the amount of liquid latex used for mixing. The masses of all four components therefore summed to the initial latex mass for each batch. The basic bismuth nitrate mass was calculated from the estimated dry rubber mass using a DRC value of 35%. Since the actual DRC was not experimentally determined, the formulation levels do not represent experimentally verified filler mass fractions relative to the actual dry rubber mass or the total mass of the dried sheets.

Table 1. Formulation masses of natural rubber sheets prepared with different basic bismuth nitrate formulation levels

Formulation level (%)	Initial latex mass (g)	Estimated dry rubber mass in initial latex (g)	Calculated basic bismuth nitrate mass (g)	PEG4000 (g)	Distilled water equivalent mass (g)	Calculated liquid latex mass for mixing (g)
0	235.00	82.25	0	20	9.98	205.02
3	236.00	82.60	2.478	20	9.98	203.542
5	224.70	78.645	3.93225	20	9.98	190.78775
7	236.70	82.845	5.79915	20	9.98	200.92085
9	235.50	82.425	7.41825	20	9.98	198.10175
11	234.10	81.935	9.01285	20	9.98	195.10715

Note: The estimated dry rubber mass was calculated using a DRC value of 35%. The actual DRC of the latex batch was not experimentally determined; therefore, these values are estimates rather than measured properties. Masses measured directly using a balance were recorded to two decimal places (0.01 g). The distilled water was dispensed volumetrically as 10 mL and represented by an equivalent mass of approximately 9.98 g in the formulation mass balance, using an assumed water density of approximately 0.998 g/mL. This equivalent mass was calculated rather than independently measured by weighing. Additional

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decimal places in the calculated formulation values represent arithmetic results and do not indicate greater experimental weighing precision.

First, 20 g of PEG4000 was mixed with 10 mL of distilled water using a magnetic hot-plate stirrer at 60 °C and 300 rpm for 1 h. Basic bismuth nitrate was then added gradually according to the calculated mass for each formulation while maintaining the temperature and stirring speed at 60 °C and 300 rpm. The resulting mixture was cooled to room temperature before being combined with the corresponding mass of natural rubber field latex. The combined mixture was manually stirred using a spatula until the components appeared visually mixed. It was then poured into an acrylic mold measuring 17 × 17 × 0.8 cm and left at room temperature to solidify. After solidification, the sheets were removed from the mold and dried for two days without direct exposure to sunlight. The drying temperature was not measured or controlled, and the attainment of constant mass was not verified. After drying, the final mass and dimensions of each sheet were measured. The resulting samples were characterized using density measurements and SEM-EDX analysis. The preparation procedure is illustrated in Figure 1.

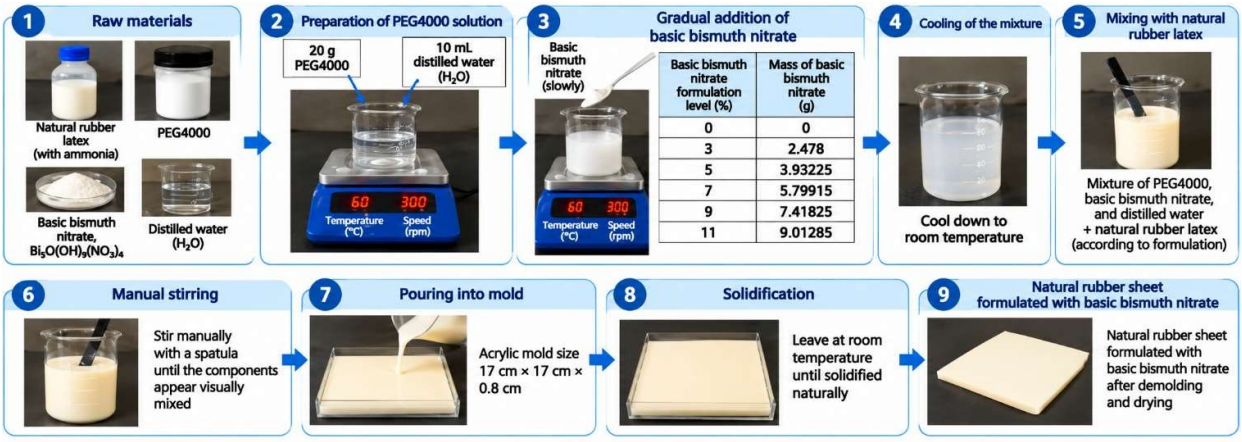


Figure 1. Schematic of the preparation of natural rubber sheets formulated with basic bismuth nitrate and PEG4000

2. Density

Density measurements were performed to characterize the physical properties of the prepared natural rubber sheets formulated with basic bismuth nitrate. The density of each dried sheet was determined from the ratio of its average final mass to its final geometric volume. A mass-to-volume approach has also been used to characterize polymer composites developed for potential radiation-shielding applications, as reported by Zen et al. [6].

The final mass of each sample was measured three times using a digital balance, with all three measurements performed on the same specimen. The average mass was then calculated using Equation (3):

$$\bar{m} = \frac{m_1+m_2+m_3}{3} \tag{3}$$

where $\bar{m}$ is the average final mass of the sample (g), while m_1, m_2, and m_3 represent the first, second, and third mass readings, respectively. These measurements represent repeated weighings of a single specimen rather than measurements of three independent specimens.

The final volume was calculated from the dimensions of each dried sheet. The length (L), width (W), and thickness (T) were measured in centimeters, and the volume was calculated using Equation (4):

$$V = L \times W \times T \tag{4}$$

where V is the final sample volume (cm³), L is the final length (cm), W is the final width (cm), and T is the final thickness (cm). The final measured dimensions were used rather than the nominal mold dimensions to account for dimensional changes

that may have occurred during fabrication and drying.

The density of each sample was subsequently calculated by dividing its average final mass by its final volume, as expressed in Equation (5):

$$\rho = \frac{\bar{m}}{v} \tag{5}$$

where ρ represents the sample density (g/cm³). The calculated density values were used to compare the physical characteristics of the sheets prepared at six basic bismuth nitrate formulation levels: 0%, 3%, 5%, 7%, 9%, and 11%.

3. SEM-EDX Analysis

SEM-EDX characterization was performed using a JEOL JSM-6510LA scanning electron microscope equipped with a JEOL JED-2300 energy-dispersive X-ray spectroscopy (EDS) system. The measurements were conducted at an accelerating voltage of 20 kV and a magnification of 1000×. All six formulation levels, including the control prepared without basic bismuth nitrate, were examined under the same measurement conditions.

To maintain consistency in the selection of observation areas, specimens were cut from the upper-right section of each sheet. Comparable surface regions were selected for SEM observation to facilitate morphological comparisons among the different formulations. The resulting micrographs were used to examine surface features and describe morphological differences between samples. Differences in image contrast were interpreted cautiously because SEM contrast alone is insufficient to establish the elemental or chemical identity of a specific surface feature.

Elemental composition was analyzed by EDX in the selected surface regions. Quantification was performed using the ZAF standardless quantitative analysis method reported by the instrument. The results were expressed as mass percentages and atomic percentages. In this study, the measured mass percentage of elemental bismuth (Bi) was used to compare the results for the 3%, 5%, 7%, 9%, and 11% formulation levels. These values represent the relative Bi content within the analyzed regions rather than the mass fraction of basic bismuth nitrate in the entire sheet. Quantitative EDX results may depend on the measurement conditions and quantification method [8,12].

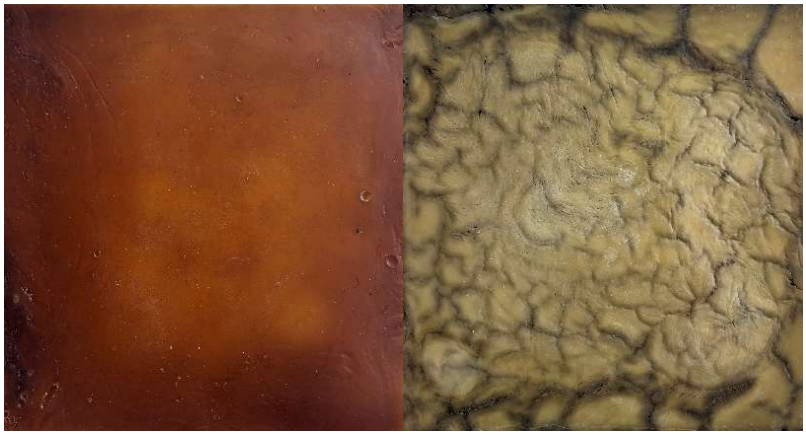
The 0% specimen served as the control because no basic bismuth nitrate was added to its formulation. Since the EDX quantification table did not report a quantified Bi mass fraction for the control, no numerical Bi value was assigned to this specimen in Figure 5; this result was not interpreted as a measured value of 0.00%. The SEM micrographs and EDX results were compared to describe differences in surface morphology and relative elemental composition among the formulations. Because EDX measurements were obtained from one selected surface region per specimen, the results characterize the analyzed local regions and do not establish uniform elemental distribution throughout the entire sheet.

III. RESULTS AND DISCUSSION

1. Physical Appearance of Natural Rubber Sheets

Natural rubber sheets formulated with basic bismuth nitrate and PEG4000 were prepared at six formulation levels: 0%, 3%, 5%, 7%, 9%, and 11%. Following molding, solidification at room temperature, and drying, the resulting materials formed sheet-like specimens with visible surface characteristics. Figure 2 compares the physical appearance of the control specimen prepared without basic bismuth nitrate (0% formulation level) and the specimen prepared at the 11% formulation level.

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(a) (b)

Figure 2. Physical appearance of natural rubber sheets formulated with basic bismuth nitrate and PEG4000 at formulation levels of 0% (a) and 11% (b)

As shown in Figure 2(a), the control sheet prepared without basic bismuth nitrate exhibits a relatively smooth and continuous surface, although minor irregularities are visible, particularly near the edges. In contrast, Figure 2(b) shows the sheet prepared at the 11% basic bismuth nitrate formulation level, which exhibits a more irregular surface characterized by visible wrinkles, cracks, and variations in texture across the observed regions. These observations indicate differences in the macroscopic surface appearance of the two formulations examined.

These differences in appearance may be associated with conditions during mixing, molding, and sheet formation. However, visual inspection alone cannot establish the specific causes of the observed wrinkles or cracks. Because Figure 2 compares only the 0% and 11% formulation levels, these observations are insufficient to determine whether surface irregularities increase progressively with increasing basic bismuth nitrate formulation level.

The macroscopic surface characteristics should be considered alongside the density measurements and SEM-EDX results. Density measurements provide information about the physical characteristics of the sheets based on their final mass and geometric volume, whereas SEM-EDX is used to examine surface morphology and identify the relative elemental composition within the analyzed regions. Together, these results provide complementary information about the physical characteristics and local elemental composition of the natural rubber sheets formulated with basic bismuth nitrate and PEG4000.

2. Density of Natural Rubber Sheets Formulated with Basic Bismuth Nitrate

Density is an important physical property for characterizing natural rubber sheets after fabrication and drying. In this study, the density of each specimen was calculated as the ratio of its average final mass to its final geometric volume. The measured density values for the six basic bismuth nitrate formulation levels (0%, 3%, 5%, 7%, 9%, and 11%) are presented in Figure 3.

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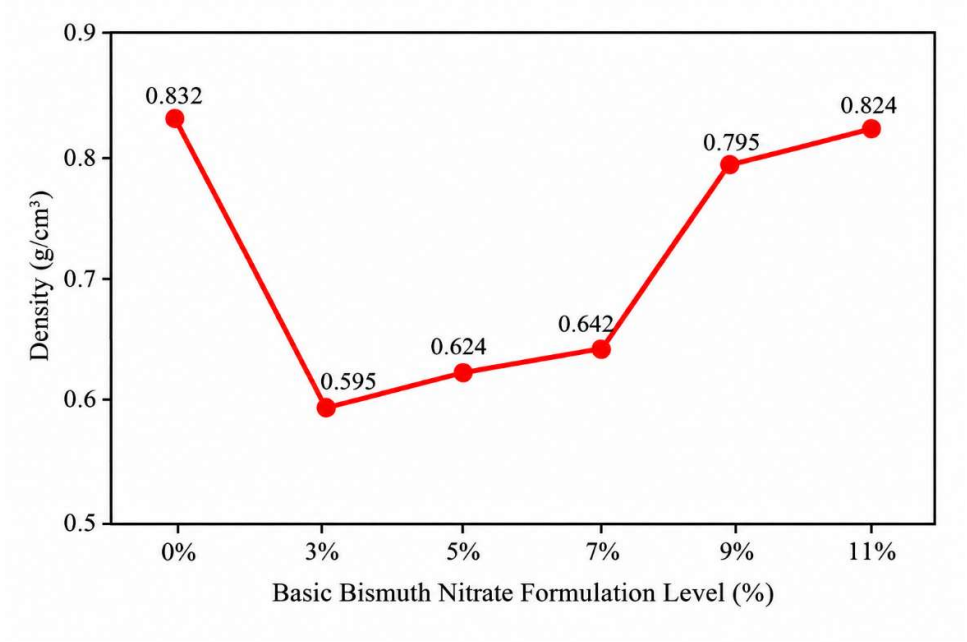


Figure 3. Measured density of natural rubber sheets at different basic bismuth nitrate formulation levels.

As shown in Figure 3, the control specimen (0% formulation level) exhibited a density of 0.832 g/cm³. At the 3% formulation level, the density decreased to 0.595 g/cm³. The density subsequently increased to 0.624 g/cm³ and 0.642 g/cm³ at the 5% and 7% levels, respectively. A further increase was observed at the 9% level, where the density reached 0.795 g/cm³. At the 11% formulation level, the measured density was 0.824 g/cm³.

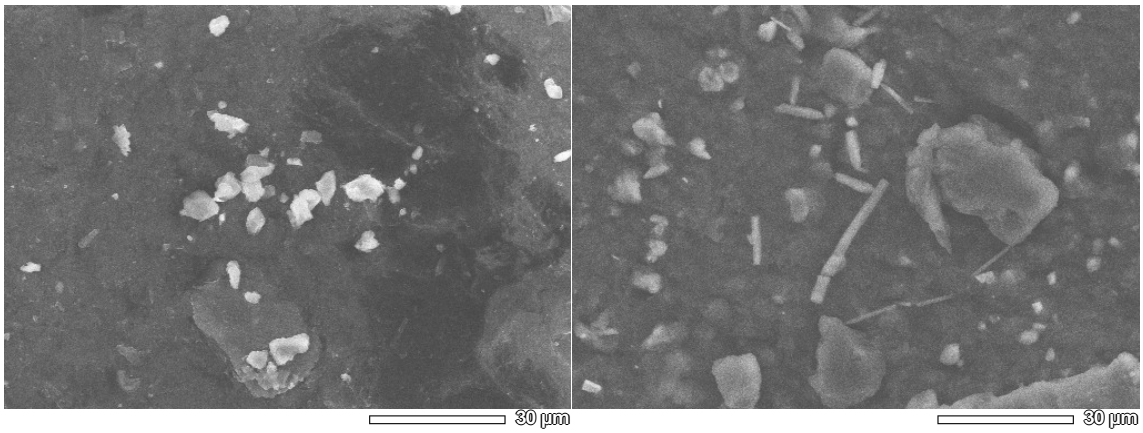
The measured density did not increase monotonically across the investigated formulation levels. The lowest density was observed at the 3% level (0.595 g/cm³), whereas the highest density among the sheets containing basic bismuth nitrate was observed at the 11% level (0.824 g/cm³). However, the control specimen exhibited a slightly higher density of 0.832 g/cm³. These results indicate that increasing the basic bismuth nitrate formulation level did not produce a consistent increase in the measured density.

The observed density differences may be associated with variations in the final mass and geometric volume of the sheets after fabrication and drying. Because density is calculated as mass divided by volume, changes in either quantity affect the calculated value. The macroscopic observations in Figure 2 also show differences in surface appearance between the control and the 11% formulation. However, these visual observations alone do not establish that surface wrinkles or cracks caused the measured density differences. Therefore, the density and morphological observations should be interpreted as complementary aspects of material characterization rather than evidence of a confirmed causal relationship.

The reported density values were calculated from the measured final mass and geometric volume of each specimen. The three mass readings used to calculate the average represented repeated weighings of the same specimen rather than measurements of three independent specimens. Consequently, the results describe the measured density of the six formulations but do not establish between-specimen variability or the statistical significance of the observed differences. In addition, the actual DRC of the field latex was not measured; therefore, the formulation levels were based on an estimated dry rubber mass rather than experimentally verified filler mass fractions relative to the actual dry rubber content. These limitations should be considered when interpreting the density results alongside the SEM-EDX findings.

3. Surface Morphology Analysis by SEM

Scanning electron microscopy (SEM) was used to examine the surface morphology of the natural rubber sheets prepared at different basic bismuth nitrate formulation levels. Figure 4 presents SEM micrographs of the control sheet (0% formulation level) and the sheet prepared at the 11% formulation level. Both images were acquired at a magnification of 1000× with a 30 μm scale bar, allowing surface features to be compared at the same magnification.



(a) (b)

Figure 4. SEM micrographs of natural rubber sheets formulated with basic bismuth nitrate at formulation levels of 0% (a) and 11% (b)

The SEM micrographs show surface features with varying shapes, sizes, and contrast levels in both samples. In the control sample, several features with brighter contrast can be observed at different locations on the examined surface. Features with irregular shapes and varying sizes are also visible in the sample prepared at the 11% basic bismuth nitrate formulation level, including regions where some features appear clustered. These observations indicate differences in the surface morphology of the examined regions. However, the SEM images alone do not establish the chemical identity of these features.

Contrast variations in SEM images alone are insufficient to identify individual bright features as basic bismuth nitrate particles. Confirming the elemental identity of a specific feature requires localized compositional information, such as EDX point analysis or elemental mapping. Furthermore, observations from the limited surface regions examined in this study cannot establish whether the filler is uniformly distributed across the entire surface or throughout the bulk of each specimen [8,12].

4. Elemental Composition Analysis

Energy-dispersive X-ray spectroscopy (EDX), performed as part of SEM-EDX characterization, was used to identify the detected elements and quantify their relative mass fractions in the analyzed surface regions. Quantification was performed using the ZAF standardless quantitative analysis method. Figure 5 presents the measured Bi mass percentages for natural rubber sheets prepared at basic bismuth nitrate formulation levels of 3%, 5%, 7%, 9%, and 11%. No quantitative Bi mass fraction was reported for the 0% control in the EDX quantification table; therefore, this sample is not included in Figure 5.

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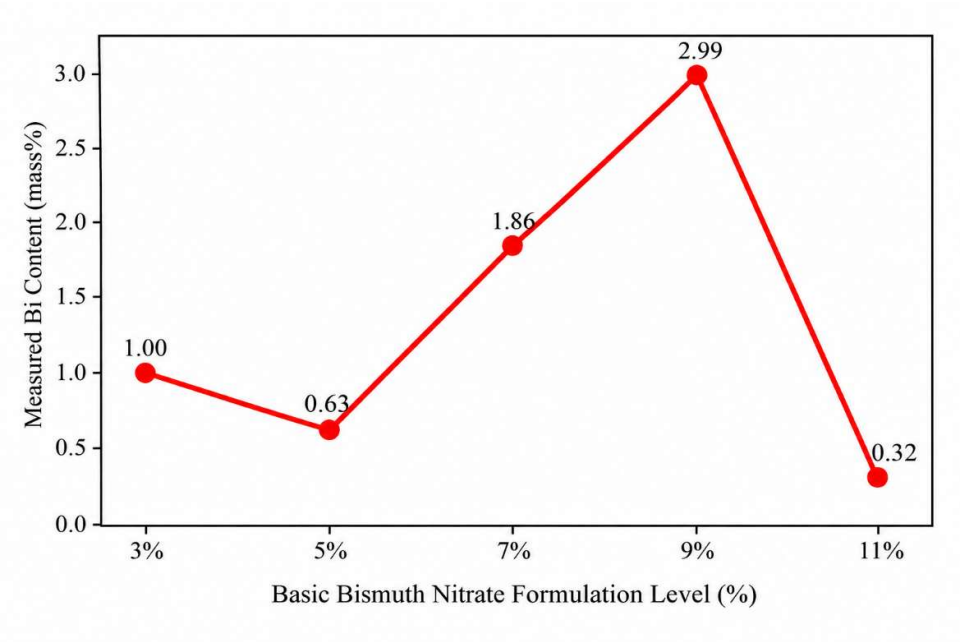


Figure 5. Measured Bi mass percentages obtained from EDX analysis of natural rubber sheets at different basic bismuth nitrate formulation levels

The EDX analysis yielded measured Bi mass percentages of 1.00%, 0.63%, 1.86%, 2.99%, and 0.32% in the analyzed regions of samples prepared at basic bismuth nitrate formulation levels of 3%, 5%, 7%, 9%, and 11%, respectively. The highest measured value was obtained at the 9% formulation level, whereas the lowest was observed at the 11% level. Thus, the measured Bi mass fraction did not increase consistently with increasing formulation level.

The measured Bi mass percentage decreased from 1.00% at the 3% formulation level to 0.63% at the 5% level, then increased to 1.86% and 2.99% at the 7% and 9% levels, respectively. At the 11% formulation level, however, the measured value decreased to 0.32%. This pattern indicates that the Bi content measured in the selected regions did not increase consistently with the formulation level. These differences should be interpreted in light of the scope and limitations of EDX measurements. The analysis was conducted on selected surface regions rather than through elemental mapping of the entire specimen. Consequently, the reported values describe the relative elemental composition of the analyzed regions and should not be interpreted as the average Bi content of the entire sheet. Local variations in elemental distribution and the limitations of quantitative EDX analysis may contribute to the observed differences [8]. Measurements from multiple regions or broader elemental mapping would be needed to assess the distribution of Bi throughout each specimen [12].

In addition to Bi, carbon (C) and oxygen (O) were detected as major elements in the EDX results for the natural rubber sheets. The measured C mass percentages across the sheets prepared at the 3%-11% basic bismuth nitrate formulation levels ranged from 60.98% to 75.75%, while the O mass percentages ranged from 20.42% to 35.61%. Other elements, including Mg, K, and Si, were also detected in some samples at comparatively lower percentages. These results describe the elemental composition within the analyzed surface regions rather than the overall mass composition of all constituents in the specimens. For the 0% control sample, the EDX quantification table did not report a quantified Bi mass fraction. Therefore, no numerical Bi value is assigned to the control sample in Figure 5. This distinguishes the absence of a quantified Bi value in the analysis table from a measured value of 0.00%.

Overall, SEM-EDX characterization revealed differences in surface morphology and measured Bi mass fractions among the analyzed natural rubber sheets formulated with basic bismuth nitrate. The highest measured Bi mass percentage was

2.99% at the 9% formulation level, whereas the lowest was 0.32% at the 11% formulation level. Because the EDX measurements were obtained from selected surface regions without elemental mapping of the entire specimens, the results do not establish the uniformity of Bi distribution throughout the sheets. Therefore, the SEM-EDX results provide preliminary information on the local surface morphology and elemental composition of the prepared sheets but do not, by themselves, demonstrate radiation attenuation performance.

IV. CONCLUSION

The preliminary characterization of natural rubber sheets formulated with basic bismuth nitrate and PEG4000 revealed variations in density, surface morphology, and local elemental composition. The measured density values at formulation levels of 0%, 3%, 5%, 7%, 9%, and 11% were 0.832, 0.595, 0.624, 0.642, 0.795, and 0.824 g/cm³, respectively. The density response was non-monotonic, with the lowest value observed at the 3% formulation level. Among the sheets containing basic bismuth nitrate, the highest density was observed at 11%, although the control sheet exhibited a slightly higher density. SEM observations revealed differences in surface features between the examined specimens. EDX analysis yielded Bi mass percentages of 1.00%, 0.63%, 1.86%, 2.99%, and 0.32% in the analyzed regions of the 3%, 5%, 7%, 9%, and 11% formulations, respectively. The highest measured Bi percentage occurred at 9%, whereas the lowest occurred at 11%. However, the actual dry rubber content (DRC) of the field latex was not measured, and the EDX measurements represented selected surface regions rather than the entire specimens. Consequently, the results do not establish the actual filler fraction relative to the dry rubber mass, the uniformity of Bi distribution throughout the sheets, or the radiation attenuation performance of the prepared materials. Further investigations, including direct DRC measurement, broader elemental mapping, and radiation attenuation testing, are required to evaluate their suitability for radiological applications.

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