

Enhancing Physical Properties of Unfilled SBR Rubber Compounds using Nano CaCO₃ Additive

Sushama Gawai¹, Sandeep Rai², Anjali Bishnoi^{3,*}

Abstract

The push for rubber compounds with improved characteristics in the rubber industry has sparked extensive research strategies for developing novel rubber compounds with improved properties like tensile strength, abrasion resistance, rolling resistance and flex properties etc., especially for the automobile industry. To intensify the processability, performance, and look of materials such as plastics, rubbers, paints, color, printing, and lubricants, additives are often employed to improve their qualities and overall value during both manufacture and application. The study explores a range of nano additives to enhance the mechanical properties of rubber composites, such as tensile strength, elongation, and resistance to wear, without sacrificing elasticity. Nano additives like calcium carbonate are low-cost additives which show improved flexibility and longevity of the rubber materials. Incorporation of such nano additives are expected to improve the reinforcing and performance properties like tensile strength, abrasion loss and rolling resistance. Improved rolling resistance is key to improve fuel efficiency of vehicles significantly. This research article is a humble attempt to investigate incorporation of readily available nano material to improvise useful properties, with a focus on cost implications. This paper investigates use of CaCO₃ nano additive (BNT900) for improving the physical and cure characteristics of rubber compounds. Overall, this research work provides a valuable insight into the application of CaCO₃ nano material in unfilled rubber compounds (without carbon black) to produce rubber materials with improved physical properties.

Keywords: Nano additives, nano CaCO₃, reinforcing properties, rubber compounds, SBR

INTRODUCTION

The increasing emphasis on sustainable development and environmental responsibility is driving change across many industries, including the rubber sector [1,2]. Traditionally, rubber production has relied on synthetic fillers, petroleum-based plasticizers, and hazardous curing agents, all of which pose significant environmental and health risks [3,4]. Conventional rubber manufacturing practices

contribute to various environmental problems, such as greenhouse gas emissions, non-degradable waste, and the release of toxic substances [5,6]. These concerns have led researchers and industry professionals to seek more sustainable alternatives. As a result, there is growing interest in developing rubber composites that not only meet or exceed existing performance standards but also reduce environmental impact [7,8]. Styrene Butadiene Rubber (SBR) is a popular synthetic elastomer because of its low cost, flexibility, and superior mechanical qualities [9,10]. Unfilled SBR compounds, on the other hand, typically have low tensile strength, poor abrasion resistance, and limited durability, rendering them unsuitable for high-performance applications [11,12]. Rubber

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composites are essential in a variety of applications, including automotive tires, seals, hoses, and footwear, where mechanical properties such as tensile strength, elongation, hardness, and abrasion resistance are critical for functionality and longevity [13,14]. Improving these properties while employing sustainable methods presents a unique challenge, requiring innovative materials and processing techniques [15].

This study aims to tackle these challenges by exploring various eco-friendly methods to enhance the mechanical properties of rubber composites. It specifically investigates the use of nano additives, bio-based plasticizers, and environmentally friendly curing agents as alternatives to more traditional, environmentally harmful materials.

Natural fillers, including bio nano additives, are becoming increasingly popular due to their biodegradability, renewability, and ability to effectively reinforce rubber matrices [16]. These fillers can improve both the mechanical strength and thermal stability of rubber composites, providing a more sustainable alternative to conventional fillers like carbon black and silica [17]. Additionally, steric acid, silica, paraffin oil and other renewable resources can enhance flexibility, processability, and durability without the toxic effects associated with traditional phthalate-based plasticizers [18].

Zinc oxide (ZnO) is a fundamental ingredient in rubber formulations, primarily because it acts as a catalyst in sulfur vulcanization, thereby shortening the curing time. To ensure proper mixing and avoid issues with dispersion, ZnO is typically introduced early in the compounding process, following the addition of stearic acid. Together with stearic acid, ZnO forms an effective activation system for sulfur-cured rubber compounds. The standard concentration ranges from 2 to 5 parts per hundred rubbers (phr) for ZnO and 1 to 4 phr for stearic acid. Additionally, smaller ZnO particle sizes tend to enhance its efficiency in the vulcanization process [19]. Stearic acid, when used in conjunction with zinc oxide (ZnO), enhances the reactivity of sulfur in the vulcanization process. The improved mechanical properties for such nanocomposites are because the applied load is efficiently transferred from matrix to the nanofillers [20].

Nanoparticles of CaCO_3 have a higher surface area, which enhances their interactions with the SBR chains in a more effective way. These will increase the interfacial bonding and improve the matrix of fillers for stress transmission [21]. This reduces the cracking and equalises the stress distribution uniformly. by preventing the formation of the crack, it leads to increased tear resistance and tensile strength [22]. Evenly spreading the CaCO_3 nanoparticles in the SBR reduces stress buildup and clumping. The nucleation during the processing produces finer and more uniform crosslinking. The load-bearing capacity with mechanical stability is improved significantly when combined with SBR and CaCO_3 nanoparticles [23].

This research also investigates the curing process and the interactions between nano additive (nano CaCO_3) within the Styrene Butadiene Rubber (SBR1502) matrix. SBR1502 is a very widely used cold and non-pigmented Styrene Butadiene Rubber with 25% bound Styrene content. A thorough understanding of interactions of SBR polymer and nano CaCO_3 is very vital for optimizing and designing of rubber formulations specially for automobile applications with optimum processing conditions to achieve superior mechanical performance. The goal of this study is to provide a detailed analysis of how low-cost nano additive material (CaCO_3) in rubber compounding can be used to produce rubber composites with a marginal cost impact. In this work, we added synthetic CaCO_3 nano additive to a Styrene Butadiene Rubber (SBR1502) compounding formula in various quantities (ranging from 0 to 9 phr) and examined its impact on cure characteristics and mechanical properties.

EXPERIMENTAL DETAILS

Materials and Methods

Calcium carbonate (CaCO_3) nano additive BNT900 was obtained from Bharat Nano Tech. Surat, India as a gift sample, synthetic rubber stylamer 1502 was received as gift sample from Reliance Industries, Vadodara, India, other chemicals sulfur, zinc oxide (ZnO), stearic acid, silica, paraffin oil,

mercaptobenzothiazyl disulfide (MBTS), tetramethylthiuram disulfide (TMTD) were gift from Naik Rubber Products, India.

Preheating nano additives

To improve the qualities of the nano additives, novel methodology employed as preheating nano additives at 120 °C for 2 hours in an oven as shown in Figure 1. The method is environmentally friendly.

Preparation of rubber compounds of SBR 1502 and BNT 900

All the rubber compounds (mixture of varying levels of BNT900 and SBR 1502) were prepared on a laboratory two roll mill and rubber compounds were processed subsequently at a roller speed of 40 rpm, and initial temperature of lab mill rolls was maintained at 40°C. Rubber compounds were made as per the recipes in Table 1. Compounds were kept for 16 hours for conditioning prior to curing. The conditioned compounds were cured on curing press at 140°C for 20 min (ASTMD 3182/89). Tensile properties were measured using a Zwick UTM Model 1445 according to ASTM D 412-92/A.

Rubber was compounded using the laboratory two-roll mill. Seven batches of rubber compounds were made with varied loadings of nano additives (BNT900) (0-9 phr). The synthesized compounds were vulcanized using a Sulphur based crosslinking method in a compression molding machine at 140°C. The compounding recipe for seven batches is tabulated below, with all constituents measured in phr (parts per hundred of rubber).



Figure 1. Preheating of the nano additives.

Table 1. Recipe of Unfilled rubber compounds of SBR1502 and BNT900 (Varying Levels).

Sr no.	Ingredients	Sample 0	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
1	Stylamer 1502	300	300	300	300	300	300	300
2	Nano additives (BNT900)	0	1.5	3	4.5	6	7.5	9
3	Zinc Oxide (ZnO)	15	15	15	15	15	15	15
4	Steric acid	3	3	3	3	3	3	3
5	Silica	150	150	150	150	150	150	150
6	Paraffin oil	15	15	15	15	15	15	15
7	MBTS	6	6	6	6	6	6	6
8	TMTD	1.5	1.5	1.5	1.5	1.5	1.5	1.5
9	Sulphur	7.5	7.5	7.5	7.5	7.5	7.5	7.5

Testing Methods

Rheological behavior (cure characteristics)

The cure parameters of uncured rubber SBR compounds were measured using a Moving Die Rheometer (MDR) at 150°C for 24 minutes. The curing curves revealed the optimal cure time (tc90), scorch time (ts2).

Mechanical and physical properties

Determination of specific gravity (SG)

The specific gravity of reclaim rubber compounds were determined by using following equation,

$$SG = \frac{M_2 - M_1}{(M_2 - M_1) - (M_3 - M_4)}$$

where, M_1 is mass of empty bottle, M_2 is mass of the bottle and dry sample, M_3 is mass of specific gravity bottle along with sample and water and M_4 is mass of specific gravity bottle filled with water only. To determine the specific gravity of the sample 50 ml of specific gravity bottle was used. For removing the moisture of the specific gravity bottle, it was kept in a hot air oven at 110 °C.

Various characterization techniques are used for the evaluation of the nanoparticle of the CaCO_3 in SBR. Each technique has its own feature and provide the particulars of various properties. It also specifies the quantitative and qualitative analysis. Scanning Electron Microscopy (SEM) study used for the surface morphology visualization and evaluate filler dispersion in to the rubber matrix, X-Ray Diffraction (XRD) analyzing the probable interactions of the of the nanoparticle of the CaCO_3 in the rubber. Transmission Electron microscopy (TEM) produces the high-resolution images at nano level to analyses particle distribution. Energy Dispersive X-ray Spectroscopy (EDX/EDS) commonly combined with SEM or TEM, allows for elemental mapping to validate the existence and uniform distribution of calcium in CaCO_3 . Moreover, Thermogravimetric Analysis (TGA) determine the thermal stability and quantitative analysis of the filler in the composites.

RESULT AND DISCUSSION

Curing characteristics of different compounds of SBR with various proportions of BNT900 loading are given in Table 2.

Optimum Cure Time (TC90) and Scorch Time

The cure characteristics and torque results are shown in Table 2. Scorch time (Ts) is the time taken for a rubber compound to begin curing (vulcanizing) at a given temperature before processing is complete. Figure 2 shows before processing rubber composite at 3 phr shows longer scorch time that the rubber remains processable during mixing without premature curing. Figure 3 shows optimum cure

time maximum at 4.5 phr shows maximum crosslinking done during vulcanization. After addition of nano additives that time was decreased. Nano additives at 3 to 4.5 phr increases the elastomeric properties of rubber composites which is a better filler for rubber composites.

Table 2. Cure characteristics of SBR Compounds with BNT 900.

phr	0	1.5	3	4.5	6	7.5	9
Optimum Cure Time (TC90)	15:54	13:10	16:36	16:59	15:20	15:32	14:26
Scorch Time (TS2)	00:45	00:38	00:59	00:03	00:24	00:27	00:30
TS1	00:30	00:14	00:31	00:12	00:09	00:10	00:11
TS5	02:57	02:07	03:00	02:01	01:40	01:54	01:32
TS10	00:33	00:22	00:35	00:20	00:11	00:17	00:15
TS50	02:09	02:57	03:52	02:59	02:06	02:49	02:28
Tensile strength	131	126	107	110	108	109	113
% at Elongation at break	542	530	520	540	503	533	523
Hardness	62	60	61	62	61	63	61
Specific Gravity	1.10	1.12	1.11	1.11	1.12	1.13	1.14

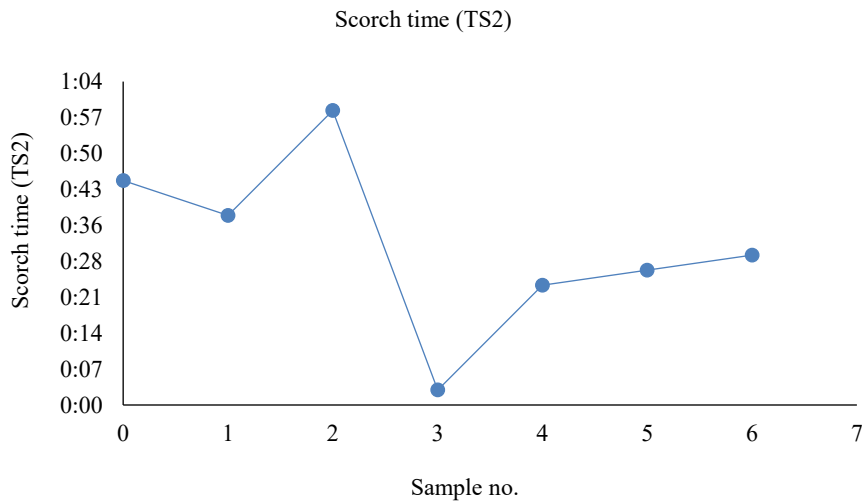


Figure 2. Effect of nano additive on scorch time.

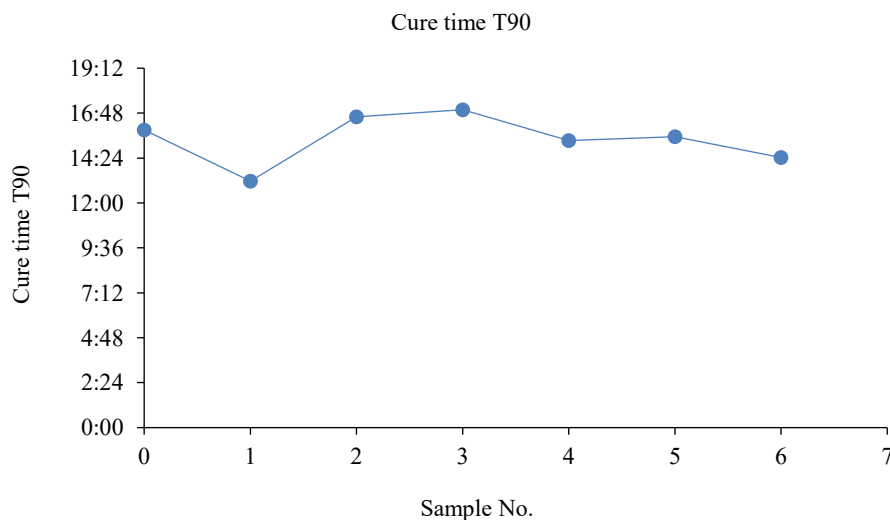


Figure 3. Effect of nano additive on optimum cure time.

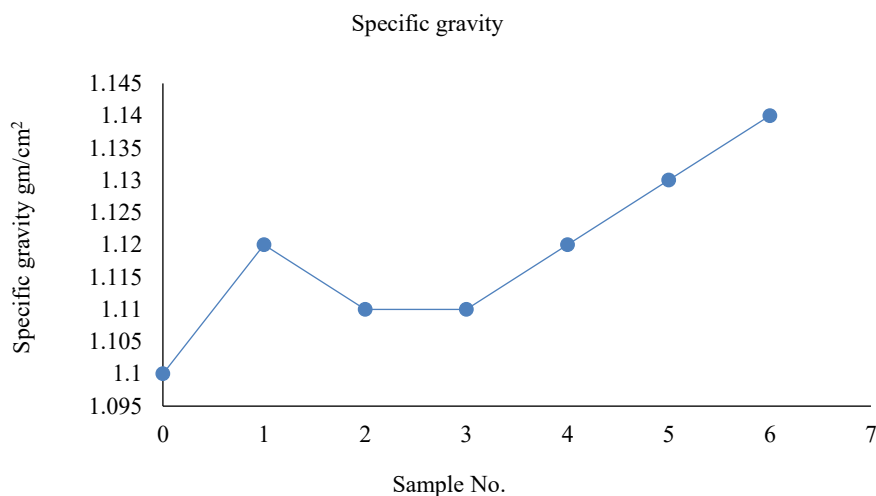


Figure 4. Effect of nano additive on specific gravity of SBR compounds.

The sample received from Bharat Nanotechnology (BNT900) has specific gravity of 2.5 gm/cm²

Specific Gravity

The specific gravity of the rubber with various proportions of the nano additives are shown in Figure 4. A comparison of values of rubber compounds indicates that there is marginal increase in specific gravity of the rubber composites with increasing nano fillers. Generally, an increase in specific gravity correlates with higher filler loading, leading to harder and stiffer rubber.

Tensile Properties

As depicted below, in Figure 5 the highest tensile strength has been observed in the sample which contains a low amount of BNT. Whereas, increasing the level of BNT in the rubber compound resulted in the lowering of the TS in most of the compounds. This phenomenon can be explained on the basis that possibly there is no interaction and bonding of nano particles with the SBR polymer matrix. However, this trend seems to be nonlinear with the varying levels of BNT nano additive. Overall, it can be concluded that the tensile strength has shown a lowering trend with increase in the proportion of the BNT in SBR rubber compounds.

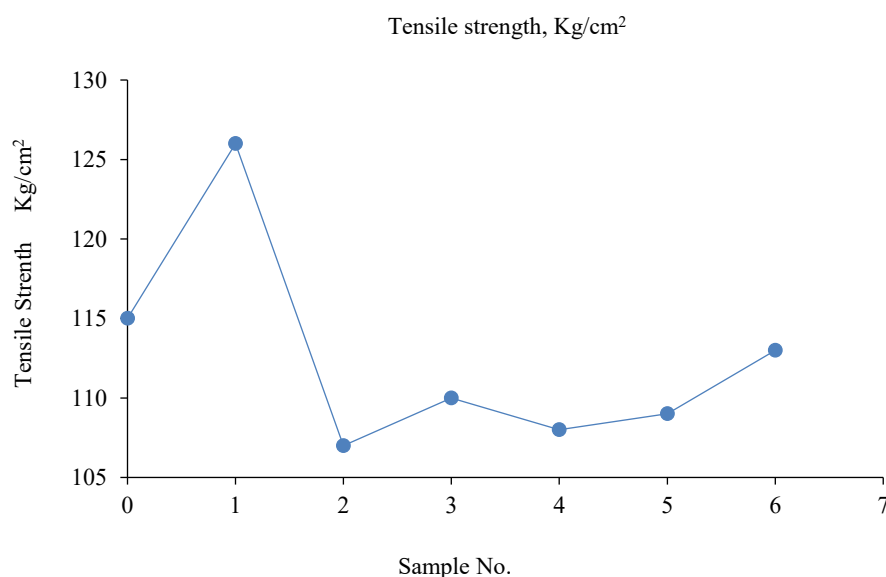


Figure 5. Effect of nano additive on tensile properties of SBR compounds.

Elongation at Break

Figure 6 depicts effect of BNT loading on Elongation at Break values of SBR rubber compounds. It can be seen from the curve that almost no improvement or increase in Elongation at break values is observed.

Hardness Shore A

Addition of nano additives increases the hardness of rubber composites. Figure 7 shows uneven trend in hardness of rubber composites with increase in nano additives. At 7.5 phr hardness is large. Increased hardness generally reduces the material's flexibility and elongation at break. Harder rubber resists abrasion and surface wear better

In SBR compounds, the incorporation of nano CaCO_3 (BNT900) affects mechanical properties due to factors such as filler-matrix interaction and dispersion. Limited interfacial bonding between nano CaCO_3 and the rubber matrix can restrict stress transfer. Surface modifications may improve adhesion, but the high surface energy of nano CaCO_3 often leads to agglomeration, resulting in stress concentration points.

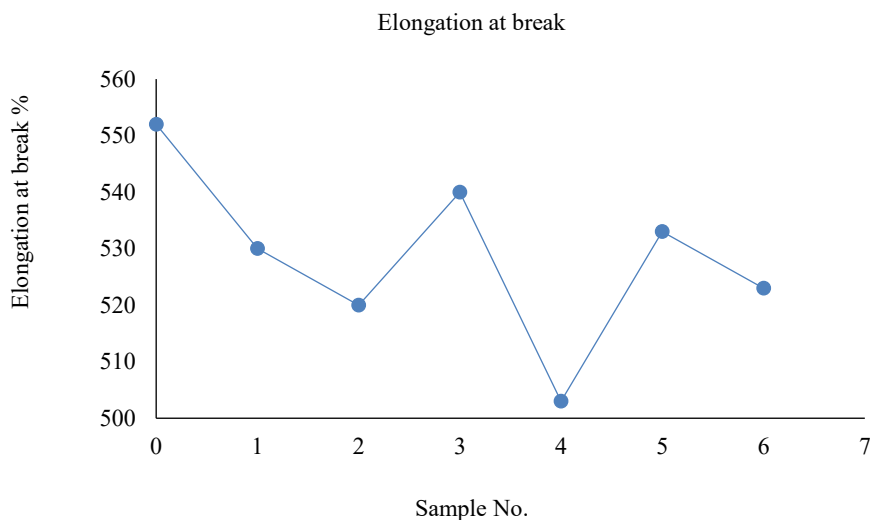


Figure 6. Effect of BNT900 loading on elongation at break % of SBR.

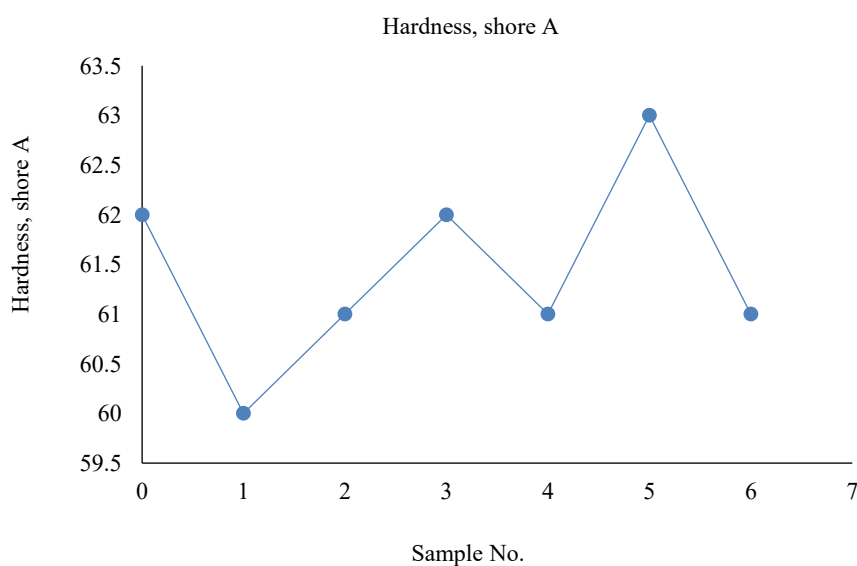


Figure 7. Effect of nano additive loading on Hardness Shore A of SBR compounds.

Compared to carbon black or silica, nano CaCO_3 possesses a lower aspect ratio and stiffness, which can reduce its effectiveness as a reinforcing filler. Its role in enhancing crosslink density during vulcanization is minimal. When present in higher amounts, nano CaCO_3 may affect the polymer network, leading to variations in tensile strength.

Compared to silica, CaCO_3 has weaker interactions with rubber chains, affecting its reinforcing potential. Identifying the optimal filler concentration is essential, as excessive, or insufficient loading may influence performance. Surface treatments, hybrid filler systems, or optimized filler content may improve the reinforcing effects of nano CaCO_3 in SBR materials

In SBR compounds, the reinforcing ability of nano CaCO_3 (BNT900) depends on its dispersion and interaction with the rubber matrix. Methods such as silane treatment, fatty acid coating, and polymer grafting help improve adhesion with SBR, allowing better stress distribution

Incorporating BNT900 with carbon black (CB) results in a combined effect that improves dispersion, bonding, and mechanical properties, optimizing tensile strength, elongation, and abrasion resistance. Advanced hybridization methods, including sol-gel coating, plasma treatment, or chemical precipitation, further enhance interfacial interactions.

Nano calcium carbonate in rubber composites offers various advantages over carbon black and silica, which also serve as effective fillers in rubber composites. Because the particle size ranges from 20 to 100 nm, it will provide the maximum surface area, which will enhance the interaction of the rubber [24]. It diffuses completely when treated with stearic acid; however, silica and carbon black require high shear mixing for uniform distribution [25]. Nano calcium carbonate in rubber composites not only retains elasticity but also increases the strength and hardness of the product. weight reduces because of low density [26]. these properties create the ideal choice for various applications in shoes, gaskets, hoses and seals. Hence, it is a low-cost alternative for the conventional composites.

The mechanical properties of rubber compounds depend on the proportion of BNT900 and carbon black (CB). A higher amount of CB enhances tensile strength, while increasing CaCO_3 content helps lower costs and adds hardness. Ensuring even distribution of fillers and choosing the right modifications is essential for better mechanical performance and ease of processing in rubber applications.

CONCLUSIONS

It was observed that the mechanical qualities of SBR vulcanizates did not exhibit significant improvement with the BNT loading from 1.5 to 9 phr. This could be attributed to the fact that SBR compounds do not contain reinforcing filler carbon black. The tensile strength and elongation at properties decreased with BNT powder content, whereas hardness and specific gravity increased marginally with increasing BNT900 loading in SBR rubber compounds as expected. This could be explained that BNT900 is nano sized CaCO_3 chemically having density of around 2.5. The findings suggest that the BNT nano additive imparts some alteration in certain physical properties and which can be used in engineering applications. Harder rubber resists abrasion and surface wear better, which is beneficial for applications like tires and industrial seals. Further plan of our research is to functionalize BNT for improving upon reinforcing properties with carbon black as reinforcing filler and use in tire tread and sidewall compounds to see the effectiveness of nano additives.

The functionalization of carbon black (CB) with nano CaCO_3 enhances rubber reinforcement by improving its interaction with SBR, distributing the filler more uniformly, and augmenting crosslink density. CB prevents the agglomeration of nano CaCO_3 , ensuring uniform distribution and enhanced stress transmission.

The substantial attraction for rubber strengthens the bond between the filler and the polymer. The fusion filler procedure enhances vulcanization, leading to increased toughness, stiffness, resistance to abrasion, and hardness. Minimizing hysteresis losses enhances tear resistance, tensile strength, and dynamic properties.

SBR, reinforced with nano CaCO₃ (BNT900), is widely used in industrial applications such as conveyor belts, pipes, and seals, where high strength, durability, and wear resistance are required. In shoe manufacturing, especially in soles, the addition of functionalized CaCO₃ and black carbon improves flexibility, wear resistance, and durability. Building materials, including sealants, adhesives, and waterproof membranes, benefit from improved mechanical properties and cost savings. Similarly, consumer goods such as sports equipment, rubber flooring, and protective gear utilize these composite materials for their strength and ease of processing.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest, financial or otherwise, related to this research.

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