



## Effect of Inorganic Blowing Agent on the Properties of Calcium Carbonate Filled Rubber Vulcanizate

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### ABSTRACT

*This study investigates the impact of sodium bicarbonate as a chemical blowing agent on various properties of natural rubber vulcanizate, with the goal of producing sponge rubber material. The experimental samples were formulated using a loading range of 0 to 12 parts per hundred of rubber (pphr), with increments of 3 pphr for the blowing agent. The sponge vulcanizates were prepared using a two-stage blowing method after mixing on a two-roll mill mixer. The first stage involved heating in an oven, followed by compression molding at 140°C for 20 minutes to obtain the sponge rubber. The inclusion of sodium bicarbonate as a blowing agent resulted in a decrease in tensile strength, hardness, and density. Tensile strength decreased from 3.93 to 3.05 MPa, hardness decreased from 45 to 33 Shores A, and density decreased from 0.90 to 0.30 g/cm<sup>3</sup>. Conversely, water uptake increased from 4.66 to 44.18%, indicating the formation of interconnected open cells. Furthermore, scanning electron microscopy revealed the formation of quasi-open cells and the presence of residual particle agglomeration of sodium carbonate due to the thermal decomposition of the blowing agent.*

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### INTRODUCTION

The interaction between the gas phase and solid matrix plays a crucial role in applications that involve unique properties such as cushioning effects, shock absorption, vibration damping, insulation, and soundproofing (Temna et al., 2014; Theerawat et al., 2022; Edmund et al., 2022). This concept is applied in the production of cellular and sponge rubber vulcanizates, where a gaseous phase is diffused and embedded into the solid rubber matrix. The gaseous phase is generated from physical or chemical blowing agents, depending on whether cellular rubber or sponge rubber is being produced, respectively (Mohammed et al., 2019; Syahrin et al., 2020).

Blowing agents play a significant role in the vulcanization process of sponge natural rubber, and common examples include N, N'

Dinitrosopentamethylenetetramine (DNPT), sodium bicarbonate (NaHCO<sub>3</sub>), and Oxybis(benzene sulfonyl) hydrazide (OBSH) (Pachareeya et al., 2013). These agents generate inert gases within the rubber matrix as temperature increases, leading to the formation of a cellular structure through thermal decomposition (Temna et al., 2014; Nuchnapa et al., 2014). Typical gases released during the foaming process include nitrogen gas (N<sub>2</sub>), carbon monoxide gas (CO), and carbon dioxide gas (CO<sub>2</sub>). The vulcanization time and temperature of rubber are vital processing parameters that influence the cellular properties achieved by the blowing agents (Worawan et al., 2014).

The thermal decomposition of the chemical agents in the rubber compound during the molding process leads to the accumulation of

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gas molecules between the rubber chains, increasing the free volume and chain mobility (Worawan et al., 2014; De Lima et al., 2021). Studies have shown that increasing the dosage of sodium bicarbonate blowing agent in the production of natural rubber latex foam results in a decrease in average cell diameter and an enhanced compression set (Sayharin et al., 2020). Temna et al. (2014) also reported on sponge rubber production using cassava starch as a blowing agent through a fermentation process, which resulted in decreased tensile strength, elongation at break, and tear strength with an increase in cassava starch content. Additionally, Najib et al. (2009) observed that the absorbed energy by natural rubber foam increases as the crosslink density and relative foam density decrease with an increased blowing agent concentration. Therefore, this article focuses on studying the effect of sodium bicarbonate on the morphology and some

physico-mechanical properties of the vulcanized rubber matrix.

## MATERIALS AND METHODS

### Materials

Natural rubber crumb, sodium bicarbonate, stearic acid, sulphur, zinc oxide, calcium carbonate, Mercaptobenzothiazole disulfide (MBTS), Trimethylquinolone (TMQ) were the major materials used in this experiment and they were supplied by Global Lab Services and Consult LTD, Nigeria. All materials were of industrial rubber processing grade.

### Methods

#### Preparation of sponge rubber vulcanizate

The experimental formulation for the preparation of test sample is based on concentration variation of sodium bicarbonate between 0 and 12 Parts per hundred of rubber (Pphr) at interval of 3 Pphr as shown in Table 1

**Table 1: Experimental Formulation (Oboh et al., 2021)**

Sample Code	Parts per hundred of rubber (pphr)							
	NR	NaHCO <sub>3</sub>	ZnO	SA	TMQ	CaCO <sub>3</sub>	MBTS	Sulphur
NRB <sub>0</sub>	100	0	5	2	1	30	2	3
NRB <sub>3</sub>	100	3	5	2	1	30	2	3
NRB <sub>6</sub>	100	6	5	2	1	30	2	3
NRB <sub>9</sub>	100	9	5	2	1	30	2	3
NRB <sub>12</sub>	100	12	5	2	1	30	2	3

NR – natural rubber, ZnO – zinc oxide, SA – stearic acid, TMQ – trimethylquinolone, MBTS – mercaptobenzothiazole disulfide, B = Sodium bicarbonate (NaHCO<sub>3</sub>) and subscript – NaHCO<sub>3</sub> concentration in Pphr

The rubber viscosity reduction and subsequent mixing with other chemicals in Table 1 were achieved in a two roll milling mixer with a friction speed of 1.20:1 at temperature of 65 ± 5 °C to obtain rubber compound. The sponge rubber formation and vulcanization was achieved via double stage method such that the rubber compound was first heated at 140 °C for 5 minutes to achieve the decomposition of the blowing agent required for expanded rubber compound formation, followed by in-situ shaping and vulcanization of test samples at temperature of 140 °C for 15 minutes

### Properties Evaluation

#### Tensile test

The tensile test was carried out using the Transcell Technology Tensometer (model BOB-200) CAP. 200 kg AT. 1.9951 MV/V according to ASTM D-638. Dumbbell shaped specimens with dimensions of 50 x 8 x 4 mm were used to perform the experiment at a loading speed of 80mm/min

#### Hardness and Density Test

Shore a hardness test for soft material was carried with the aid of the

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Durometer with Model No. 5019 accordance with ASTM D 785-08 while densities of the samples were determined by ASTM D-792.

#### Water Absorption

The water absorption was carried in accordance with ASTM D750-95. Equal-dimension of samples was immersed in water for a period of 48 hours to obtain percentage of water absorbed by various samples.

#### Scanning Electron Microscopy

The fracture surface of neat sample (NRB<sub>0</sub>) and the sample with 6pphr (NRB<sub>6</sub>) were characterized using SEM JOEL JSM-7600

### RESULT AND DISCUSSION

#### Tensile Strength

The result of tensile strength is presented in Fig. 3.1

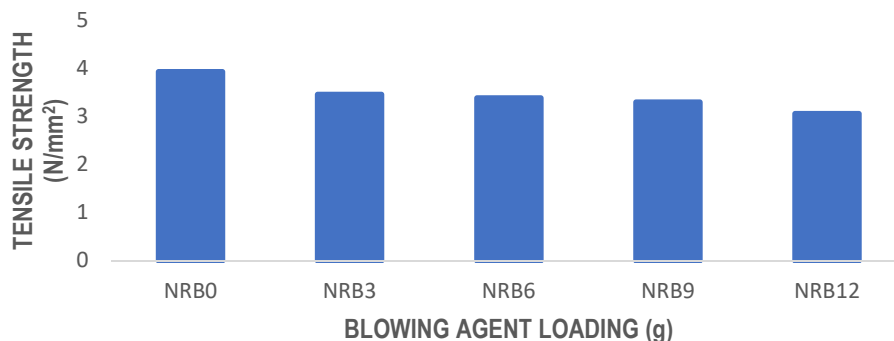


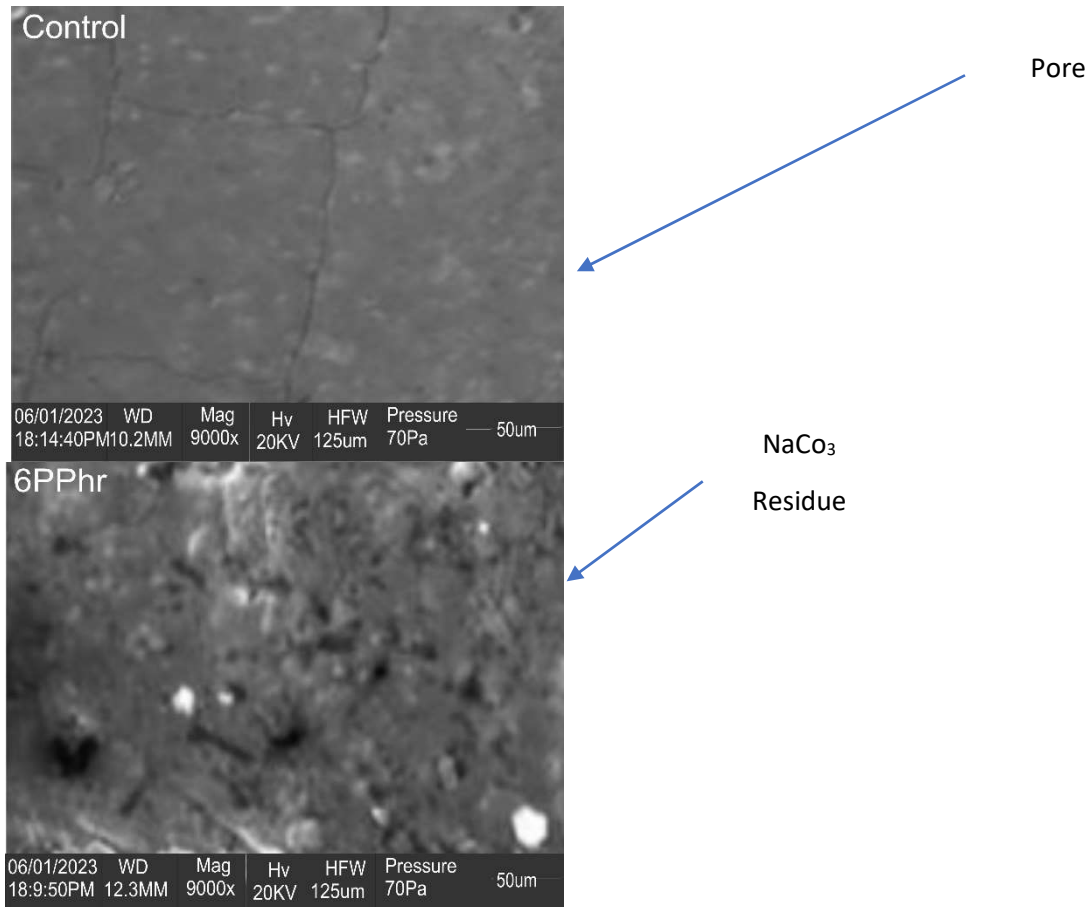
Fig 3.1: effect of blowing agent on tensile strength

The result clearly reveals a gradual reduction in tensile strength with increase in sodium bicarbonate concentration in the formulation such that it inclusion of 3, 6, 9 and 12 pphr into rubber compound give corresponding tensile strength values of 3.457, 3.378, 3.293 and 3.052 N/mm<sup>2</sup> or MPa respectively as against 3.927N/mm<sup>2</sup> or MPa for sample without blowing agent. The decrease in tensile strength can be attributed to the presence of cells or air pockets created by generation of carbon dioxide gaseous phase as a result of thermal decomposition of sodium bicarbonate; this gaseous phase is subsequently trapped within the molecules or

structure of vulcanized rubber matrix. The presences of these cells or air pockets limit the capacity to transfer external stress by the sponge rubber samples there reducing the tensile strength. Similar observations has been reported by Nasruddin and Susanto 2019

#### Scanning Electron Microscopy (SEM) Result

The SEM microgram of fractured surface of pure solid rubber vulcanizate samples without blowing agent and that of sponge rubber formulated with 6pphr of sodium bicarbonate as blowing agent are compared in Fig 3.2



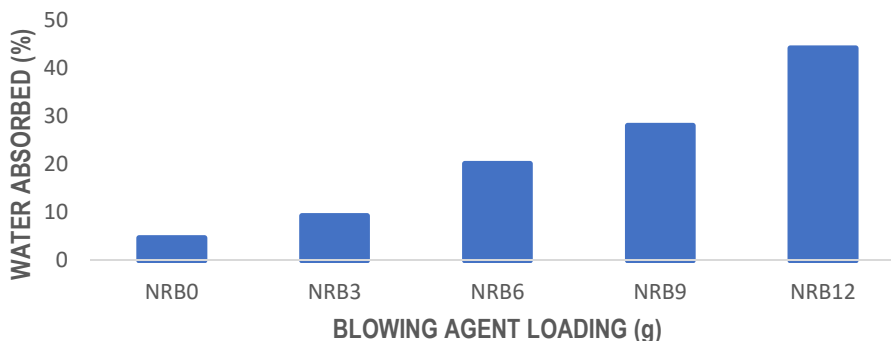
**Fig 3.2:** SEM Images of Solid Rubber and 6pphr Sponge Rubber

The pure rubber matrix without blowing agent coded control in the microgram shows relatively smooth surface without obvious presence of pores. Conversely, the sponge rubber sample formulated with 6 pphr of blowing agent presents a surface that is characterized by several open cells or pores as indicated by an arrow. Also observed are pockets of agglomerated particles in form of white patches on the microgram of the sponge rubber sample. Recall that during the sponge rubber formation which occur alongside vulcanization reaction, the sodium bicarbonate ( $\text{NaH}(\text{CO}_3)_2$ ) decomposes to

yield sodium carbonate ( $\text{NaCO}_3$ ), carbon dioxide and water, under the condition of vulcanization the gaseous phase of water and carbon dioxide are given off leaving behind a residual solid of sodium carbonate within the vulcanizate matrix. Hence the agglomerated particles are most likely sodium carbonate.

#### **Water Absorption**

The result of the percentage water absorbed under the specified conditions is shown in Fig 3.2



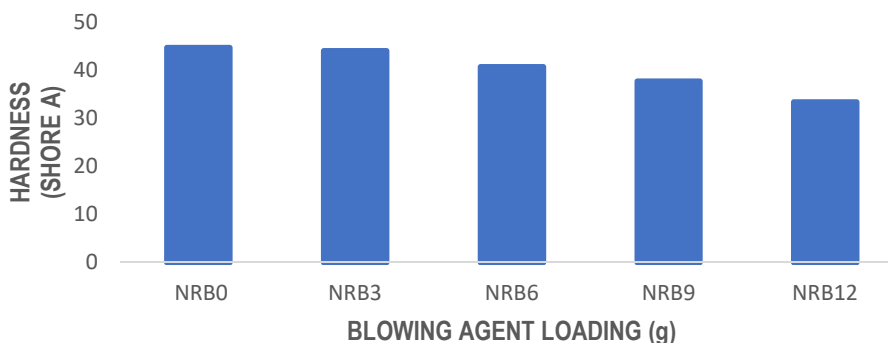
**Fig 3.3:** Effect of blowing agent on the Water Absorption

It is observed from the result that percentage of water absorption is increasing with increasing concentration of blowing agents such that 4.65, 9.25, 20.13, 28.05 and 44.18 % of water were absorbed by samples containing 0, 3, 6, 9 and 12 pphr of blowing agent respectively. This is expected as increase presence of blowing culminate to higher ratios of cells or air pockets, therefore, these cells can serve as holding vessels for fluids such as water and hence the

reason for the observed trend. Furthermore these phenomenon suggest the formation of continuous open cell in the rubber matrix (Pachareeya *et al.*, 2013).

**Hardness**

Shore a hardness scale is used for soft materials in the category of this experimental samples hence the results obtained from its application is shown in Fig 3.4



**Fig 3.4:** Effect of blowing agent on the hardness

The hardness of rubber samples is the resistance to permanent surface indentation. The trend of decreasing hardness with increase in concentration of blowing is again observed here. The presence of air pores trapped within the rubber molecules resulted to softer matrix

material there by making it less resistance to indentation. (Worawan *et al.*, 2013)

**Density**

The densities of various samples of the sponge rubber produced is given in Fig 3.5

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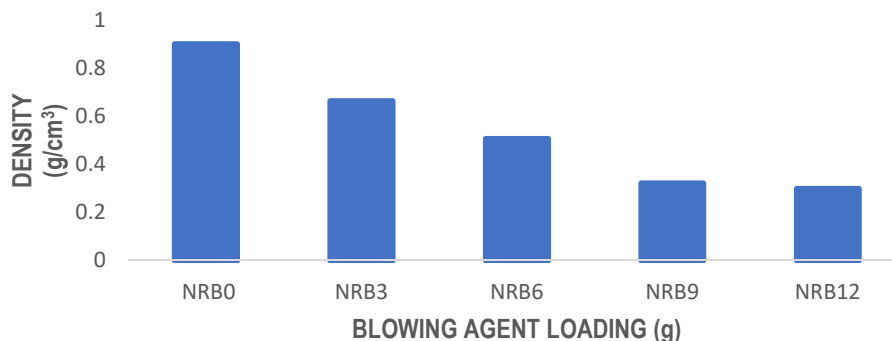


Fig 3.5: Effect of blowing agent on the density

The result showed that the density decreased with increasing blowing agent loading content. The sponge rubber samples formulated with 3, 6, 9 and 12 pphr of blowing agent had 0.664, 0.507, 0.322, and 0.299 g/cm<sup>3</sup> respectively as against pure solid rubber matrix with a density of 0.901 g/cm<sup>3</sup>. The decreased density properties of the rubber sample with increased percentage blowing agent loading could be related to the fact that blowing agent (NaHCO<sub>3</sub>) in the rubber matrix may soften the rubber core making it less dense. Similar trend is observed by (Najib *et al.*, 2022) research, where they carried out an investigation on the effect of blowing agent concentration on cell morphology

### CONCLUSION

The preparation of sponge rubber using natural rubber and sodium bicarbonate as a blowing agent was successfully carried out. The result obtained showed that tensile strength, hardness, density and water resistance decreased with increasing concentration of sodium bicarbonate. The scanning electron microscopic study reveals the formation of open cell in the vulcanized rubber matrix.

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