The effect of sodium bicarbonate ratio for the mechanical properties of underarm pads rubber for crutches

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Abstract. Research on the effect of the ratio of sodium bicarbonate on the mechanical properties of underarm crutch pads from natural rubber SIR-20 has been carried out. The ratio of sodium bicarbonate used was 1.00 phr; 1.50 phr; 2.00 phr; and 2.50 phr. The rubber compound for underarm crutch pads was made by the compounding process using an open rubber mixing mill and vulcanization at a temperature of 120 ºC for 11 minutes. The best formula for curing characteristic rheometer test results at a temperature of 150 ºC was URC-04 with \(S_{\text{max}}= 4.95 \text{ kg/cm}, S_{\text{min}}= 0.44 \text{ kg/cm}, \Delta S = 4.51 \text{ kg/cm}, t_{\phi} = 6.57 \text{ min} \) and \(t_{t2} = 4.13 \text{ min}\). The mechanical test result showed the hardness test before aging and after aging of 31 Shore A, and 32 shore A, tear strength of 8.50 kN/m, elongation at the break before aging and after aging of 300%. The results of the identification of functional groups by FTIR for infrared absorption in each sample generally showed the same pattern with one another. SEM analysis result showed the distribution of materials and the formation of pores by sodium bicarbonate was not evenly distributed with an average diameter of 5 microns.

1. Introduction
Underarm rubber is a soft rubber formed by air bubbles that are trapped between its molecules. Underarm rubber is placed at the top of the crutch as a base to feel comfortable when touching the underarm rubber when used. Crutches are aids for patients who have temporary or permanent health problems especially for walking [1-2]. Materials that are widely used to make underarm rubber for crutches generally come from polyurethane rubber, this is due to lightweight polyurethane rubber with a specific gravity of 36 kg/m³. Polyurethane resin compositions using polyols have an average molecular weight below 400 [3]. Polyurethane is a synthetic rubber from urethane compounds (ONHCOO), widely used for various products because it has high thermal stability, is resistant to weather, ozone, and radiation [3-4]. Polyurethane made from polyisocyanates, polyhydroxy compounds, and a small number of macromolecular compounds -NHCOO is widely used as an organic polymer material for various purposes such as medical equipment, textile industry, building construction, and so on [5].

The development of polyurethanes for various purposes such as for underarm rubber has been carried out by many industries and researchers. Latinwo [6] developed polyurethane foam with fillers CaCO\(_3\) and CaMg(CO\(_3\))\(_2\). According to Chen [7] using micro and nano-size CaCO\(_3\) fillers can increase foam cell density. Agarry [8], and Usman [9] developed polyurethane foam with CaCO\(_3\) fillers. Marhoon [10] used a filler from rice husk ash as a polyurethane filler. Underarm rubber made of polyurethane, based on observations and interviews with users can easily be torn. Tearing can be
caused by the structure of the wall between the pores formed by carbon dioxide gas trapped in polyurethane molecules that do not have a strong bond.

Based on the description above, this study used solid natural rubber of the SIR-20 type as an ingredient in underarm rubber. Natural rubber is a hydrocarbon polymer that is naturally elastic in the form of a white liquid such as milk [11]. Natural rubber with the general formula of isoprene (C₅H₈) and with the chemical structure of cis-1,4-polyisoprene, which is not easy to tear, has widely been used for various applications [12-14]. The development of natural rubber into underarm rubber in this study was carried out by adding blowing agent materials, fillers, softeners, and additives.

Blowing agent materials that are widely used to form air cavities into foam are categorized as organic and inorganic materials. Organic blowing agents are for example dichloromethane, freon, pentane [15], while inorganic blowing agents contain elements such as carbon dioxide, nitrogen, and air [16]. The material used to form air cavities in rubber vulcanisate is sodium bicarbonate which is commonly used by industry and researchers. The air cavity formed in the rubber is a polymer foam that has good elasticity, which will return to its original state when under pressure from other objects.

Polymer foam has advantages including, low density, good heat insulation, can be used as a damper, and it has corrosion resistance [17]. According to Hussein [18], the reason for using sodium bicarbonate as a foam material is because the price is cheap and does not pollute the environment. Mohammad [19], used sodium bicarbonate as a blowing agent with a ratio of 4 phr that produced a density of 0.9 with water absorption by cells 1.7 x 10⁻³ g/min, and maximum compression of 105 kPa. Ariff et al. [20] used sodium bicarbonate as a foam development agent at vulcanization temperatures of 140, 150, and 160 °C. Najib [21], developed rubber foam using sodium bicarbonate in a ratio (4, 8, 10, and 12) phrase, and the results showed that the increase in sodium bicarbonate could increase the cell volume. Cell volume increased due to the presence of air that came out to form the cell nucleus permanently from the nucleation [22-23].

The development of natural rubber into underarm rubber used zinc oxide activators and stearic acid as co-activators. The activator was used to accelerate the performance of the accelerator during the vulcanization process. Wardana [24] used ZnO and stearic acid as activators and stearic acid as co-activators with results that could increase the efficiency and rate of the air vapor transmission. Duan [25] studied the effects of ZnO nanoparticles and stearic acid in the manufacture of foam rubber with the result of increased tear strength and tensile strength.

Fillers were used in making foam to strengthen the structure of cell wall networks and reduce production costs. Tangboriboon et al. [26] used 60 phr CaCO₃ fillers with NaHCO₃ as a blowing agent that produced a bulk density of 240.5 kg/m³, a relative foam density of 0.2614, cross-link density of 74.279 mol/m³, the compression set of 32.60%, and thermal conductivity of 0.066 W/mK. This study used a multi filler consisting of kaolin, CaCO₃, and zeosil. Kaolin and CaCO₃ are fillers to reduce production costs, and zeosil to improve the mechanical properties of underarm rubber. According to Rosszainily [27], calcium carbonate as a filler can improve the thermal properties of rubber vulcanisate. Zeosil is amorphous precipitated silica that is more easily dispersed in rubber compounds. Zeosil is widely used as a tire filler to reduce hysteresis of compounds and heat buildup while maintaining rigidity. To improve the homogeneity of fillers and additives in the rubber matrix structure, minarex oil was added as a softener [28]. This research studied the effect of sodium bicarbonate ratio by using multi fillers (kaolin, CaCO₃, and zeosil) with softener minarex oil on the mechanical properties of the underarm rubber vulcanisate. To determine the maturity of the underarm rubber compound, a curing characteristic rheometer test was performed. The mechanical properties of rubber vulcanisate tested were hardness before aging and after aging, tear strength, elongation at the break before and after aging.

Functional group analysis was done using Fourier transform infrared (FTIR). The uniformity of material distribution and formation of pores by sodium bicarbonate were observed using a scanning electron microscope (SEM). This research is expected to provide information on the development of natural rubber as a raw material for underarm rubber material along with sodium bicarbonate as a blowing agent, and multi-fillers consisting of kaolin, CaCO₃, and zeosil.
2. Materials and methods

2.1. Materials

The materials used in this research included SIR-20 natural rubber. Titanium dioxide (TiO₂) MBR-9672 purity 94% min, oil absorption 19 g/100 g max. Pigment & dyestuff [22094-93-5] pigment yellow 81, density 94% min, heat resistance 180 °C, light fastness 7, water resistance 5, oil resistance 5, and alkali resistance 5. Yipin inorganic pigment powder S-130 iron oxide red for color paving brick max moisture 1%, color difference 1.0, predominant particle 0.1-0.3, main purity 99%, water (oil) absorption 15-25%, density 5.0 g/m³. Zinc oxide (ZnO) purity 99.70%, water-soluble ≤ 0.10%. Stearic acid purity 99% min. Kaolin 325 mesh from Tanjungpandan Belitung Indonesia. Calcium carbonate (CaCO₃) purity 98%, pH 9.5%, whiteness 97% min. Zeosil 175P. Pacific rubber processing oil (paraffinic) E-245 density 0.88 g/mL @ 29.5 °C, pour point, -6 °C, aromatics 10%, napthenic 30%, paraffin 60%, density 0.88 g/ml @ 29.5 °C, pour point, -6 °C, aniline point, 96 °C, viscosity @ 40 °C 30-32 CST. Sodium bicarbonate (NaHCO₃) purity 99% min, density 2.16 g/mL at 25 °C (lit.). Vulcanization accelerator MBTS DM/2,2-dithiobis (benzothiazole)120-78-5 purity 98%, ash 0.3%. TMTM (TS) accelerator with the chemical formula C₆H₄N₂S₂ yellow powder the specific density was 1.37-1.40. Rubber chemicals PVI/rubber retarder anti scorching agent PVI. Rubber vulcanizer insoluble sulfur/OT20 use in tyre purity 99%, ash max 0.15%, fineness (100 mesh) ≥ 99.8%, oil content oil content 20±1%.

2.2. Methods

2.2.1. Mastication process and manufacture of rubber compound . The development of natural rubber into underarm rubber in this study consisted of 4 (four) formulas. Each formula was differentiated based on the ratio of sodium bicarbonate used. Natural rubber for all treatments was weighed according to the ratio as shown in table 1.

| Material     | URC-01* | URC-02* | URC-03* | URC-04* |
|--------------|---------|---------|---------|---------|
| NR           | 100.00  | 100.00  | 100.00  | 100.00  |
| Titanium     | 5.00    | 5.00    | 5.00    | 5.00    |
| Pigment Yellow | 6.00   | 6.00    | 6.00    | 6.00    |
| Pigment Red  | 0.25    | 0.25    | 0.25    | 0.25    |
| ZnO          | 5.00    | 5.00    | 5.00    | 5.00    |
| Stearic Acid | 2.00    | 2.00    | 2.00    | 2.00    |
| Kaolin       | 45.00   | 45.00   | 45.00   | 45.00   |
| CaCO₃        | 10.00   | 10.00   | 10.00   | 10.00   |
| Zeosil       | 12.50   | 12.50   | 12.50   | 12.50   |
| Paraffinic Oil | 22.00 | 22.00   | 22.00   | 22.00   |
| NaHCO₃       | 1.00    | 1.50    | 2.00    | 2.50    |
| MBTS         | 1.00    | 1.00    | 1.00    | 1.00    |
| TMTM         | 0.50    | 0.50    | 0.50    | 0.50    |
| PVI          | 0.01    | 0.01    | 0.01    | 0.01    |
| Sulfur       | 2.00    | 2.00    | 2.00    | 2.00    |

Annotation: *phr = parts per hundred of rubber
URC = underarm pads rubber for crutches

After each rubber was weighed according to the material ratio in table 1, the mastication process was carried out through milling using an open mill type RSK-300. Next, after mastication (± 15 minutes) titanium oxide was added to make white rubber so easy to stain. After the rubber turned white, color pigments were added (red and yellow composites) until the whole rubber turned yellow. The next process was the addition of ZnO and stearic acid to improve the performance of the
accelerator in the vulcanization process. Then kaolin, CaCO$_3$, zeolite were added also at the same time as paraffinic oil as a softener and sodium bicarbonate until the mixture became homogeneous. While continuing milling, MBTS, and TMTM as accelerators were added at the same time as PVI/rubber retarder was added. The final process of compounding was adding sulfur as a vulcanizing agent. Each formula was replicated three times to get the best data. Table 1 is the material ratio of each treatment.

2.2.2. **Compound vulcanization process.** The compounds of each formula were vulcanized and molded at 120 °C for 11 minutes

2.2.3. **Testing of rubber compound and rubber vulcanizates.** Rubber compound testing was carried out to determine the maturation characteristics of rubber compounds into vulcanizates. The rheometer was used according to ASTM 5289. The test of mechanical properties included hardness (ASTM D.2240-15), elongation at break (ASTM D.412-16), tear strength (ASTM D.624-00 (ra2012)), and ozone resistance at 25 pphm, 20% (D.1149 -16). Identification of functional groups was done using the Agilent array 630 FTIR spectrometer, wavenumber range 7,000-350 cm$^{-1}$ /5,100–600 cm$^{-1}$, interferometer 60° air bearing interferometer, data integrity such as 21 CFR Part 11 and EU Annex 11. The Fourier transform infrared (FTIR) spectroscopy was used to study functional groups. The identification of the surface of the objects was conducted using scanning electron microscopy (SEM) JSM-6510 LA with the magnification 5 times up to 300,000 times (on 128 mm x 96 mm image size).

3. **Results and discussion**

3.1. **The curing characteristics of rubber compounds**

Characteristics of rubber compound maturation for underarm crutch rubber from 4 (four) samples are shown in table 2.

| Curing Characteristic | Rheometer 150 °C | Test Result |
|-----------------------|------------------|-------------|
|                       | URC-01 | URC -02   | URC -03 | URC -04 |
| S$^*$ Maximum, kg-cm  | 4.93   | 5.12       | 5.00    | 4.95    |
| S$^*$ Minimum, kg-cm  | 0.66   | 0.36       | 0.54    | 0.44    |
| $\Delta$s (Kg/cm)     | 4.27   | 4.76       | 4.46    | 4.51    |
| Opt cure time (t$_{90}$) min; sec | 8.00 | 10.26 | 8.52 | 6.57 |
| Scorch time (t$_{67}$), min; sec | 4.16 | 6.9   | 5.24   | 4.13    |

Note: = Test Method PPK, Using MDR 2000 (Not accredited)

Characteristics of compound maturation for underarm crutch rubber are generally explained by the results of testing maximum torque (S$_{max}$), minimum torque (S$_{min}$), delta torque ($\Delta$s) ($S_{max}$-$S_{min}$), optimal maturation time (t$_{90}$) and scorch time (t$_{67}$). The maximum torque test of rubber compounds for underarm crutch rubber in this study was to study changes in the viscosity value of each difference in the addition of sodium bicarbonate. The change of viscosity was related to the formation of air cavities due to the performance of sodium bicarbonate which released CO$_2$ gas into the rubber compound during vulcanization.

The CO$_2$ gas released in the rubber compound from the decomposition of sodium bicarbonate during vulcanization affected the formation of cross bonds and the molecular structure of the underarm rubber vulcanisate. According to Najib [21], cross-linking and foam density are relatively decreased with the energy absorbed by the foam, and at the same time, the formation of foam cells and the increase in the number of cells per unit area also occurs. As shown in Table 2, the highest maximum torque value (5.12 kg-cm) was obtained at the sodium bicarbonate ratio of 1.5 phr (table 1), while the highest minimum torque value (0.66 kg-cm) was observed at URC-01 with sodium bicarbonate ratio 1 phr.
Viewed from an increase in the sodium bicarbonate ratio, an increase in the maximum torque value and non-uniform minimum torque fluctuated with an increase in the sodium bicarbonate ratio. This might be due to the performance of sodium bicarbonate releasing CO$_2$ gas into the rubber compound matrix when vulcanization occurred. This event resulted in the disruption of the formation of a cross-linking degree during vulcanization. The highest value (4.76 kg/cm$^2$) degree of crosslinking formed during vulcanization occurred at a sodium bicarbonate ratio of 2.0 phr (URC-02).

For an optimal maturation time (10.26 minutes) at a sodium bicarbonate ratio of 1.5 phr (URC-02), the maturation before the vulcanization process lasted for 6.6 minutes with a sodium bicarbonate ratio of 1.5 phr (URC-02) with a value of 6.9 minutes. According to Poh [30], a ratio of sodium carbonate exceeding 1.5 phr can increase the speed of vulcanization, but there is a reduction in the scorch time and cure index. The time of maturation and the formation of cross-link during the vulcanization process are influenced by the ratio of sodium bicarbonate. Besides, the speed of maturation of rubber compounds and the number of pores formed by CO$_2$ affect the strength of the cross-linking formed.

3.2. Mechanical properties of vulcanisate
The test results of the mechanical properties of underarm crutch rubber vulcanisate are shown in figures 1, 2, and 3.

![Figure 1. Effect of material ratio to hardness](chart.png)

Figure 1 shows the results of the underarm rubber hardness from 4 formulas. The hardness values for the URC-01 and URC-03 before aging had the same value (30 Shore A), for the URC-04 formula, the value increased to 31 Shore A. The highest hardness value before aging and after aging was obtained at URC-02. The difference in the value of hardness illustrates the difference in the level of material density in the rubber vulcanizates matrix per mm$^3$.

The level of space density in the rubber vulcanisate matrix was determined by the material ratio (table 1), the distribution of materials, and the vulcanization process. In addition, the effect of sodium bicarbonate which formed air cavities by CO$_2$ gas affected the density and structure of molecules that made up vulcanisate rubber. Sodium bicarbonate was a blowing agent that produced CO$_2$ and H$_2$O when it received heat [31]. The reduced level of crosslinking density in the molecular structure of the rubber compound resulted in a better and more homogeneous formation of foam [32]. The amount of CO$_2$ gas trapped between vulcanisate rubber molecules was inversely proportional to the hardness value.

The elongations at break test results for the URC-01 formula before and after aging were 220%, and 210.94% respectively, it was the lowest compared to other formulas (figure 2). The differences in the value of the elongation at break were due to the number of air cavities that were formed reduced the density of spaces and bonds between molecules. According to Roy [33], sodium bicarbonate
during heating decomposes into sodium carbonate, water, and CO$_2$. CO$_2$ gas trapped in the polymer matrix produces foam. The amount of CO$_2$ gas formed reduces the strength of the double bond, which in turn decreases the value of the elongation at break. This is because the empty space formed by CO$_2$ gas has no molecular bonds. The elongation at break value is also influenced by additives such as ZnO. Where the ratio of additives added will affect cross-linking. The ZnO ratio at 5 phr showed a significant reduction in tensile strength, elongation at break, modulus, hardness, and compression [34]. Meanwhile, if the ZnO ratio is more than 7 phr, there will be a decrease in tensile strength, elongation at break, modulus, and hardness [35]. In this study ZnO ratio for all formulas was 5 phr, but the ratio of ZnO to elongation at break was also influenced by the ratio of sodium bicarbonate and other additives (table 1).

The elongation at break value in the URC-02 formula before aging was 360%, while after aging was 348.89%. It was the highest compared to other formulas. As seen from the sodium bicarbonate ratio of 1.5 phr and the filler ratio (table 1), it was possible that in this ratio (URC-02) the amount of CO$_2$ formed was proportional to the amount of filler available. Whereas in the URC-04 formula with a sodium bicarbonate ratio of 2.5 phr, the elongation value before aging and after aging was the same, which was 300%.

![Figure 2. Effect of material ratio to elongation at break.](image)

In the formula URC-04, there was a balance of the amount of CO$_2$ and the availability of fillers so that the value of elongation at the break before and after aging was also the same. In figure 2 there is a decrease in the value elongation at break from URC-02 to URC-04. An increase in the ratio of sodium bicarbonate decreases the elongation value. Elongation at break is a stretch that occurs when the object is pulled in the opposite direction at the critical point experiencing a break [34]. This is because the bonds formed between material molecules do not have the ability to resist the forces acting on them so that when a strain occurs it breaks.

Tear strength as well as elongation at break is one of the measuring parameter to see the resistance of objects when experiencing bonding movements between molecules until the critical point is torn. The tear strength is the force required to tear a sample, the stronger the bonds between molecules, the higher the tear strength [36]. The tear strength test result for the URC-02 formula was 10.5 kN/m, the highest compared to the other formulas. Meanwhile, for the URC-01 tear strength value was the lowest compared to other formulas (figure 3). Tear strength is influenced by the strength of the cross-linking, where the energy required is directly proportional to the strength of the materials that form the cross-link [37].

The difference in tear strength is also influenced by the amount of air that is present between the material molecules. The amount of air trapped is influenced by the ratio of the constituent material
The amount of CO$_2$ as a blowing material in underarm rubber in this study was influenced by the ratio of sodium bicarbonate and other materials that made it up. The amount of CO$_2$ formed in the underarm rubber vulcanisate in the same space when there is a difference will affect bonds between molecules. In addition, the amount of CO$_2$ formed in this study affected the density of the cross-linking formed by sulfur.

![Figure 3. Effect of material ratio to the tear strength](image)

The number of air bubbles trapped reduced the density of the rubber vulcanisate. Low cross-linking density affected tear strength. With the weak cross-linking that formed underarm rubber vulcanisate, the tear strength also became weak. The cross-linking formed was influenced by the ratio of the activator and accelerator. According to Mariano et al. [38], mechanical properties are affected by the amount of accelerator added. Tear strength is the amount of force used to stretch the bond between the material molecules to the critical point until the tear.

3.3. Fourier Transform Infrared
Observation of samples was conducted using Fourier transform infrared (FTIR) and scanning electron microscope (SEM) to determine the wavenumber, chemical composition, and surface morphology of the sample [39]. The results of the characterization of vulcanisate rubber by FTIR using sodium bicarbonate showed a number of bonds formed. The bonds formed were C - H atomic bonds with different peaks in each treatment (table 1) (figures 4, 5, 6, and 7).

![Graph showing spectral analysis](image)

The graphs in Fig 4, 5, 6, and 7, show the same pattern between one graph and another. The main functional group are represented by wave number i.e. 3600 cm$^{-1}$ (Uptake of the OH group); 3000 cm$^{-1}$ (absorption of aliphatic C-H); 1525 cm$^{-1}$ (uptake of the -COO group); 1000 - 1100 cm$^{-1}$ (absorption of Si-O interactions); <750 cm$^{-1}$, fingerprint area according to the functional group identification table. However, based on data from the graph above it can be seen that there was a spectral difference between the four formulas. At wavelengths between 1000 cm$^{-1}$ to 1100 cm$^{-1}$ there is a Si-O-Si bonding group in which at each spectral result, in each different formula, a line formed a valley.

The silica detected in the graph above was silica added as a filler to strengthen the cell wall structure between the spaces formed by CO$_2$ from the decomposition of sodium bicarbonate by heat. Silica sources detected on the graph came from kaolin, CaCO$_3$, and zeosil (table 1).

3.4. Morphological properties of vulcanize underarm rubber
Testing of rubber vulcanisate was done using scanning electron microscopy (SEM) to determine the distribution of air cavities that occur by sodium bicarbonate in the rubber compound matrix, and the bonds among substances used [29]. Scanning with SEM was carried out at 5000x magnification as shown in figures 8, 9, 10, and 11.
Figure 4. URC-01 axillary rubber vulcanisate wave number.

Figure 5. URC-02 axillary rubber vulcanisate wave number.

Figure 6. URC-03 axillary rubber vulcanisate wave number.

Figure 7. URC-04 axillary rubber vulcanisate wave number.

Figure 8. Enlargement of URC 01 underarm rubber morphology with SEM 5000x.

Figure 9. Enlargement of URC 02 underarm rubber morphology with SEM 5000x.
SEM analysis showed that the morphological differences were influenced by the ratio of sodium bicarbonate and additives used. The results of morphological testing using SEM with a magnification of 5000x showed that the air cavity of each sample was not uniform in size. The air cavity occurred due to the interaction between the rubber compound phase and the blowing agent [40]. In addition, the formation of non-uniform air cavities from each sample was influenced by the ratio of the addition of the blowing agent [41].

The results of morphological scanning with an electron microscope on underarm rubber vulcanisate samples using SEM in figures 8, 9, 10, and 11 with 5000 times magnification were intended to see the air cavity formed by CO$_2$ in rubber vulcanisate resulting from the decomposition of sodium bicarbonate by heat. From the pictures above it can be seen that there were differences in the air cavity formed between the vulcanized rubber molecules and additives. The air cavity formed by CO$_2$ in the picture above was a hole with a non-uniform diameter. The difference in the distribution of the blowing agent in each part during the formation of foam by CO$_2$ gas resulted in the formation of different air cavities [42]. This was due to the distribution of CO$_2$ in the underarm rubber vulcanisate that was not homogeneous, also there was a possibility that fillers and additives added were not homogeneously distributed.

Thus, the effect of the distribution of material influenced cross-linking by sulfur by producing non-uniform double bond strength. As a result, air cavities were formed with varying diameters. In figure 8 air cavity was formed between 1.021 $\mu$m – 934.6 nm, in figure 9 air cavity was formed between 1.290 nm - 836.7 nm, in figure 10 air cavity was formed between 1.021 $\mu$m - 772.1 nm, and in figure 11 air cavity was formed between 1.802 $\mu$m - 983.3 nm. In general, the diversity of air cavities formed by CO$_2$ on average was 5 $\mu$m.

4. Conclusion
The time of maturation and the formation of cross-link during the vulcanization process were influenced by the ratio of sodium bicarbonate. In addition, the speed of maturation of rubber compounds and the number of pores formed by CO$_2$ affected the strength of the cross-linking formed. The amount of CO$_2$ gas trapped in the rubber compound matrix was influenced by the ratio of sodium bicarbonate and the density of filler added. The amount of CO$_2$ formed also affected the mechanical properties of vulcanisate rubber. Besides, based on the results of tests using FTIR there were differences in functional groups, and based on the results of scanning with SEM there were differences in the diameter of the air cavity and the distribution of fillers from each treatment.
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