

EFFECT OF COMPOUND FORMULATION ON THE PRODUCTION AND PROPERTIES OF EPOXIDISED NATURAL RUBBER (ENR-25) FOAMS

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In this study, Epoxidized Natural Rubber (ENR-25) formulations are compounded and tested to obtain a stable expandable rubber foam as well as to determine the foam cell physical morphology and its mechanical properties. The experiment was carried out by employing different ratio of rubber blend between ENR-25 and natural rubber (SMR-L), different amount of blowing agent which is Sodium Bicarbonate and different ratio of accelerator between Tetramethylthiuram-disulfenamide (TMTD) and N-cyclohexyl-2-benzotiazolsulfenamide (CBS). Cure characteristics of the compounded rubber were determined using Monsanto Rheometer and the expandable rubber foam is produced using compression moulding technique with utilization of heat transfer process. The generated foam cell morphology is analyzed using image analyzer and SEM while the mechanical properties of the foam are examined by using Instron machine to determine its compression stress. On the other hand, swelling test using the Flory-Rhener equation is also done to support the substitute results. The data shows that at the ratio of 60 phr ENR-25 and 40 phr SMR-L is able to deliver stable rubber foam. Furthermore, increasing the amounts of blowing agent evidently increased the foam cell size, inducing smaller cell in between the foam cell wall, decreasing the value of compression stress and compression set. It was also observed that increasing the ratio of CBS in the compounded rubber will increase the cell size, creating thicker foam cell wall, increasing the value of compression stress and compression set.

Introduction

To date, there has been great development in the making of foam product and mostly for the applications of cushioning to make mattress, house whole used and others. Beside these applications, foam also has a good demand for engineering applications such as packaging product, thermal insulations for buildings and also in automotives industries. The main advantage of foam over other type of materials is its low density which leads in creating lightweight product. Furthermore, the production of foam goods itself covers 10% of total usage of polymer materials and generally 70% of the foam products are made using polyurethane followed by polystyrene and polyvinyl chloride [1].

Although thermoplastic are well known features in producing a foam, thermoset and elastomer also has been used to produced a foam but its rarely commercialize due to lack of research done. Latex foam is most favourable materials since it provides properties in elasticity hence contribute in a lot of general applications. But unfortunately, to preserve the latex milk in a liquid form before going through other process, it involves in adding an amount of ammonia to prevent it from coagulation and technically ammonia is hazardous to the environment. For elastomer, the challenge to make foam is contributing to its high viscosity and tedious formulations. Formulations play a very important role in altering the overall properties of

the generated foam and sometimes mistakable or not compatible formulations will lead to cell collapse or even no foam are produce [2]. Different amount and type of blowing agent used will effect the overall cell growth and distributions of the cell growth of the foam. There has been research done to study the rubber foam but most of them utilized combination of two blowing agent, mostly between ADC and sodium bicarbonate [3,4].

There limited literatures that covers the usage only sodium bicarbonate due to low volume of carbon dioxide release during the decomposition. Therefore, in this particular study only sodium bicarbonate is put to the test to evaluate its capability in promoting expansion to the rubber compound. Besides that, various types of formulations are also formulated to determine the change to the rubber foam.

Experimental

Materials and Formulations

ENR-25 with 25 mol% epoxidation and SMR-L was supplied by Malaysian Rubber Board. The vulcanization additives such as Zinc Oxide (ZnO), silica S, stearic acid, Tetramethylthiuram-disulfenamide (TMTD), N-cyclohexyl-2-benzotiazolsulfenamide (CBS) and sodium bicarbonate were obtained from Bayer (M) Sdn. Bhd. Whereas, sodium bicarbonate was

purchased from Merck, Inc. All materials were used as supplied and the complete formulations are shown in Table 1,2 and 3

Table 1: Formulation for different ratio of rubber blend between ENR-25 and SMR-L

Formulation	phr		
ENR-25	100	80	60
SMR-L	-	20	40
Na ₂ CO ₃	8	8	8
TMTD	2.5	2.5	2.5
CBS	1	1	1
Sulphur	0.5	0.5	0.5
Stearic Acid	2	2	2
Silica	5	5	5
Zinc Oxide	4	4	4

Table 2: Formulation for different amount of sodium bicarbonate

Formulation	phr		
ENR-25	60	60	60
SMR-L	40	40	40
Na ₂ CO ₃	4	6	8
TMTD	2.5	2.5	2.5
CBS	1	1	1
Sulphur	0.5	0.5	0.5
Stearic Acid	2	2	2
Silica	5	5	5
Zinc Oxide	4	4	4

Table 3: Formulation for different ratio of accelerator between TMTD and CBS

Formulation	phr			
ENR-25	60	60	60	60
SMR-L	40	40	40	40
Na ₂ CO ₃	8	8	8	8
TMTD	2.5	2.0	1.5	1.0
CBS	1.0	1.5	2.0	2.5
Sulphur	0.5	0.5	0.5	0.5
Stearic Acid	2	2	2	2
Silica	5	5	5	5
Zinc Oxide	4	4	4	4

Mixing and Cure Characteristic Measurement

The rubber compound was compounded with other ingredients based on the formulations of the mixture in a two-roll mill; model XK-160, in accordance with ASTM method designation D 3184-80. The curing agent which is sulphur and the blowing agent were added at the end of the process. The cure characteristics of the compounded rubber were determined by using a Monsanto Rheometer model MDR 2000 at 150°C.

Sample Preparation

The rubber compound is moulded in the disc-shaped with the diameters of 160 mm and 6 mm depth using the hot press for 1 minute at the temperature of

100°C. After moulding, it was directly transferred into an oven set at a temperature of 150°C for 30 minutes. The generated expandable compound was then taken out from the oven and allowed to cool down under room temperature.

Morphological Study

Studies on morphology of various formulations foam were carried out using a scanning electron microscope (SEM), model Leica Cambridge S-360. Utilizing the obtained SEM micrographs, image analyzer software, Image Pro Plus, is used to obtain the average cell size produced parallel and perpendicular to the foaming direction. The results of average cell size were determined from measurement at 20 different locations of the captured image. The cell amount was calculated from the knowledge of average cell size, solid rubber density and foam density.

Measurement of Physical Properties

The crosslink density test was studied in toluene according to ASTM D 471-98 using the Flory-Rhener equations. To find the density of the solid vulcanized rubber without expansion, the test was done based on ASTM D1817-96. Relative foam density was measured using equation in accordance with ASTM D3575-93. Whereas, the foam expansion ratio was calculated based on the following equation.

$$\text{Expansion ratio} = \frac{H_f - H_o}{H_o}$$

where,

$$\begin{aligned} H_f &= \text{Height after foaming} \\ H_o &= \text{Initial height before foaming} \end{aligned}$$

Measurement of Mechanical Properties

Sample with the dimension of 50x50x23 mm was cut without any skin layer exists on the top and bottom of the rubber foam. Then, it was tested using Instron machine with a cross head speed of 25 mm/min downwards to measure its compression stress. For compression set, the sample with the dimension of 50x50x18 mm was cut and put through the compression plate and compressed until 50% of the foam original thickness before exposing it in an oven with the temperature of 70°C for 24 hours. The percentage of the compression set is calculated using the equations below.

$$\text{Compression set} = \frac{t_o - t_r}{t_o - t_c} \times 100\% \quad (1)$$

$$\begin{aligned} t_o &= \text{Initial thickness} \\ t_r &= \text{Thickness after recovery} \\ t_c &= \text{Thickness after compression set} \end{aligned}$$

Results and Discussion

The Effect of Different ratio of Rubber Blend between ENR-25 and SMR-L

Rheology

The rheograph results between the three compounds with different ratio of rubber blend between SMR-L and ENR-50 shows more or less similar patterns as shown in Figure 1.

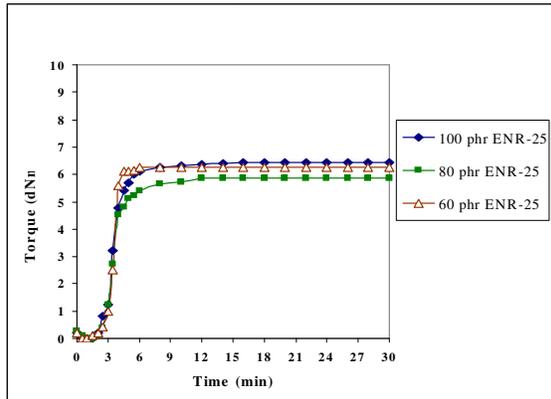


Figure 1: Rheograph for different blend ratio of rubber compound between ENR-25 and SMR-L

Regarding the data generated in Table 4,

Table 4

Cure characteristic data for different blend ratio of rubber compound between ENR-25 and SMR-L

Properties	100 phr ENR-25	80 phr ENR-25	60 phr ENR-25
M_L (dNm)	0.01	0.17	0.09
M_H (dNm)	6.48	5.87	6.26
Optimum cure torque, T_{90} (dNm)	5.63	5.38	5.57
Optimum cure time, t_{90} (min)	5.44	5.34	4.92
Scorj time, t_2 (min)	1.93	1.97	1.89
Minimum torque time, t_1 (min)	0.88	0.93	0.91

the optimum cure time T_{90} exhibit higher value with increasing ratio of ENR-25. According to Ismail and coworkers [5] in their research on the cure characteristic for the blend of NBR and ENR-50, proves the cure characteristic rose slightly with adding more content of ENR-50. Basically the experimental blend for SMR-L and ENR-25 seems to follow similar trend. These particular phenomena happen due to the activation of crosslinking on epoxy group attached to the backbone of the ENR-50 rubber chain.

As for M_H which is the maximum torque, evidently the viscosity for 80 phr ENR-25 had lessen thoroughly compared to the corresponding 100 phr ENR-25 and 60

phr ENR-25. This is similar to findings reported by Ismail and Leong [6] for SMR-L blend with Chloroprene Rubber (CR). This scenario occurred due to lower viscosity value of the SMR-L than that of CR which results in occurrence of an interphase between the two rubber blend which created incompatible blend. In these cases, similar situation can be projected to exist with the rubber blend between ENR-25 and SMR-L.

Foaming Process

From the observation after 1 hour of foaming process, foam produced from the blend of 60 phr ENR-25 proved to be a stable foam instead of 100 phr ENR-25 and 80 phr ENR-25 as shown in Figure 2.

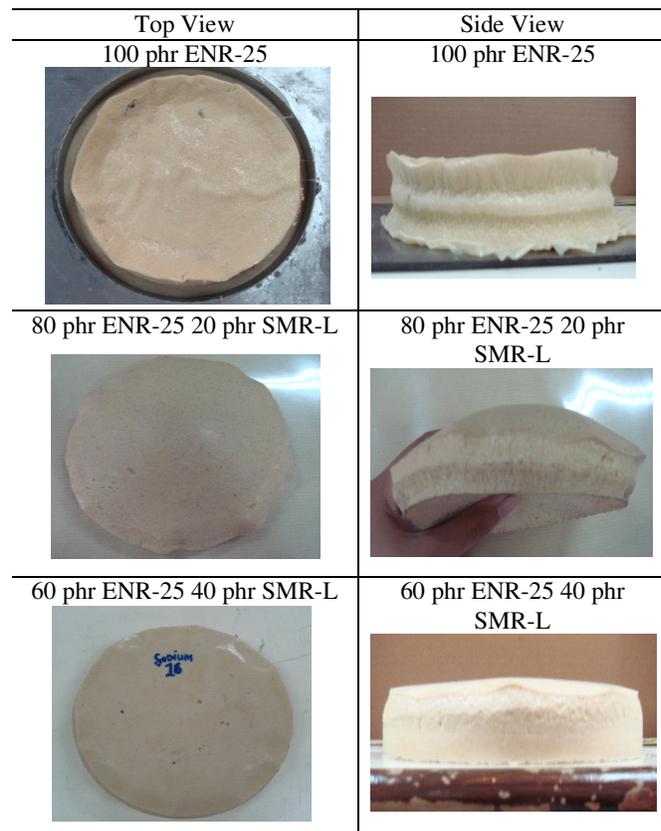


Figure 2: The view of different blend ratio of rubber compound between ENR-25 and SMR-L after foaming.

Referring to Landrock [7], foaming process will go through two stages which are expansion on the early stage and followed by shrinkage. Expansions are caused by the thermal degradation of the blowing agent while shrinkage occurs when blowing agent has decomposed completely. The cure characteristic data shown in Table 5

Table 5

Cure characteristic data for different blend ratio of rubber compound between ENR-25 and SMR-L

Properties	100 phr ENR-25	80 phr ENR-25	60 phr ENR-25
M_L (dNm)	0.01	0.17	0.09
M_H (dNm)	6.48	5.87	6.26
Optimum cure torque, T_{90} (dNm)	5.63	5.38	5.57
Optimum cure time, t_{90} (min)	5.44	5.34	4.92
Scorch time, t_2 (min)	1.93	1.97	1.89
Minimum torque time, t_1 (min)	0.88	0.93	0.91

provides details where as the amount of ENR-25 increases, the cure time t_{90} will evidently decrease. Since the formulation with 60 phr ENR-25 cured faster compared to 100 phr ENR-25 and 80 phr ENR-25 these revealed that the crosslinking has already completed when the blowing agent has fully decomposed for 60 phr ENR-25. Whereas, formulations with 100 phr ENR-25 and 80 phr ENR-25 took longer time to complete the crosslinking. Consequently after the blowing agent had completely decomposed, the cell wall is not fully cured and strong enough to uphold the rubber mass and slowly, it will collapse with time. This can be clearly seen in Figure 2.

The Effect of Adding Different Amount of Sodium Bicarbonate

Rheology

The research was continued with the effect of adding different amount of sodium bicarbonate. Since the usage of 60 phr ENR-25 are capable of producing a stable foam without any shrinkage, so the formulation are adapted for these study and the amount of blowing agent introduced are altered. Based on the rheograph generated in Figure 3.

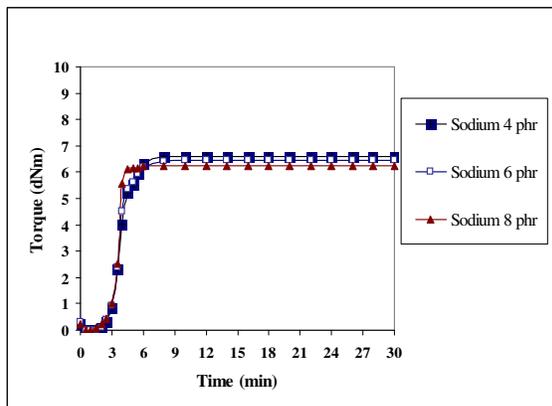


Figure 3: Rheograph for different amount of sodium bicarbonate

It can be clearly observed that all the compounded rubber shows slight differences. From the rheograph value of M_H , t_2 , T_{90} and t_{90} seems reduce with adding more content of sodium bicarbonate. Previously, Ariff [8] has investigated on the viscosity of the compounded foam concluded that increasing amount of blowing agent into the compounded polymer will result in releasing more volume of gas. Normally for sodium bicarbonate, majority of the gas release is carbon dioxide. Carbon dioxide consist small molecules which allowed it to easily absorb into the compounded rubber and reduce the matrix viscosity. As a result, it directly affects the crosslinking rate due to higher volumetric gas decomposition in the vulcanized rubber. The data is supported by the crosslink density results shown in Figure 4

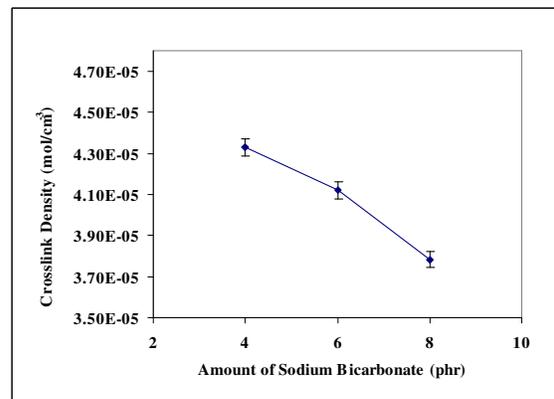


Figure 4: Crosslink density for different amount of sodium bicarbonate

which also show increasing value of crosslink density with increasing content of blowing agent.

Morphology and Physical Properties of the Rubber Foam

There are various factors influencing the foam morphology such as temperature, pressure, type of polymeric materials and the formulation used. The effect of chemical blowing agent introduces will definitely effect the cell distribution and the cell size depending on the type of blowing agent used, loading amount and the blowing agent particle size [2].

The investigation also observed that, the cell size grow larger when higher amount of sodium bicarbonate regardless of foaming direction, i.e. perpendicular or parallel and this is shown in Figure 5.

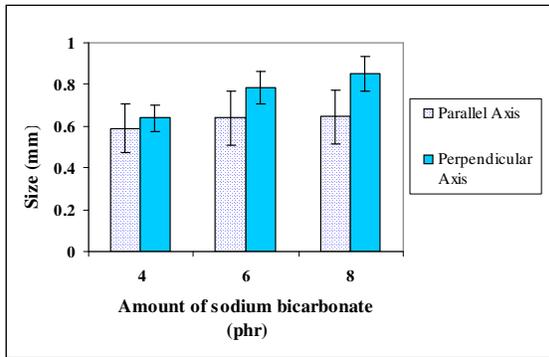


Figure 5: Cell size for different amount of sodium bicarbonate

The cause can be related to reduction in matrix viscosity due to partially complete crosslinking and consequently produced larger cells with increasing amount of blowing agent. A graphical representation of this occurrence can be seen in Figure 6.

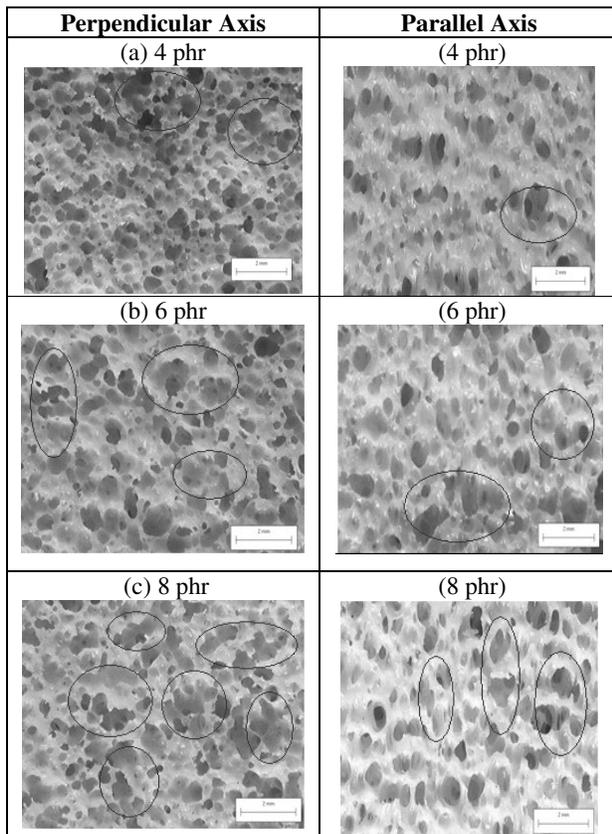


Figure 6: Cell distributions for different amount of sodium bicarbonate

Increasing cell size and thinning effect of the cell wall will directly alter the foam density as well as foam expansion ratio. Relatively, the foam density is reduced and the ratio of foam expansion will also increase.

On the other hand, the foam relative density is highly influenced by the chemical crosslinking

generated among the polymer chain. The crosslinking will form a network consisting of higher packing polymer chain and results in increasing foam relative density (refer Figure 7).

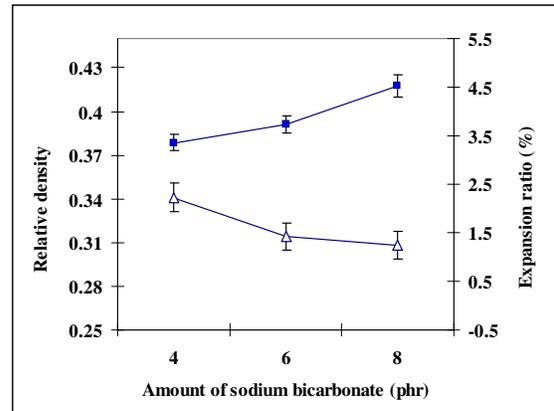


Figure 7: The effect of relative density and the ratio of foaming expansion for different amount of sodium bicarbonate

Again, the data is supported by the calculated cell amount which is tabulated in Table 6.

Table 6
Cure characteristic data for different amount of sodium bicarbonate

Properties	4 phr Sodium	6 phr Sodium	8 phr Sodium
M_L (dNm)	0.11	0.07	0.09
M_H (dNm)	6.52	6.48	6.26
Optimum cure torque, T_{90} (dNm)	5.86	5.72	5.57
Optimum cure time, t_{90} (min)	5.22	5.04	4.92
Scorch time, t_2 (min)	1.56	1.68	1.89
Minimum torque time, t_1 (min)	0.81	0.93	0.91

The result shows that the amounts of cell decrease with increasing amount of sodium bicarbonate. Logically, the cell size will increase when more open cells are produced. The data are strongly connected to Figure 6 which showed the foam morphology present more open cell exist as increasing amount of sodium bicarbonate.

Mechanical Properties

Physical data of its physical properties will directly or indirectly affect the foam mechanical properties. As for compression set, it is determined that by adding more sodium bicarbonate, less value of compression set are obtained. According to Ariff and colleagues [9], the cell size plays an important role in compression set test.

Normally the bigger cell will collapse first rather than the smaller ones due to microscopic deformation at the cell wall but the results are in reverse direction as shown in Figure 8.

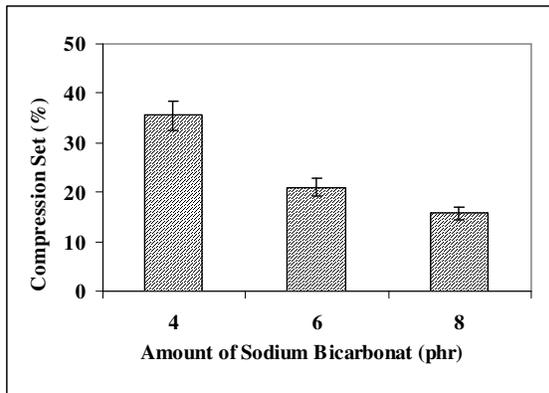


Figure 8: Compression set for different amount of sodium bicarbonate

Supposedly, bigger cell sizes are prone to undergo microscopic deformation rather than the smaller size. But, in this situation it is possible that during the deformation the smaller cells in between the cell wall were not damaged or ruptured and recovered to give a better recovery for the foam.

Generally, as the cell morphology changes, the foam will also give different value of compression stress. It is discovered that the compression stress is reduced with increasing content of sodium bicarbonate as shown in Table 7.

Table 7
Data for compression set for different amount of sodium bicarbonate

Rubber foam	Sodium bicarbonate (4 phr)	Sodium bicarbonate (6 phr)	Sodium bicarbonate (8phr)
Compression Modulus (kPa)	0.8792 ± 0.008	0.7125 ± 0.011	0.5451 ± 0.021
Compression stress at 25% strain rate (kPa)	17.598 ± 0.024	14.687 ± 0.031	12.707 ± 0.018
Compression stress at 50% strain rate (kPa)	46.09 ± 0.017	46.51 ± 0.009	34.24 ± 0.014
Maximum strain (%)	69.13 ± 0.012	67.93 ± 0.014	66.66 ± 0.009

Bigger cell size and thinner cell wall will reduce the resistant towards bending. As a consequent, it is observed that foam produced with 8 phr of sodium

bicarbonate displayed the lowest value followed by 6 phr and 4 phr as shown in Figure 9.

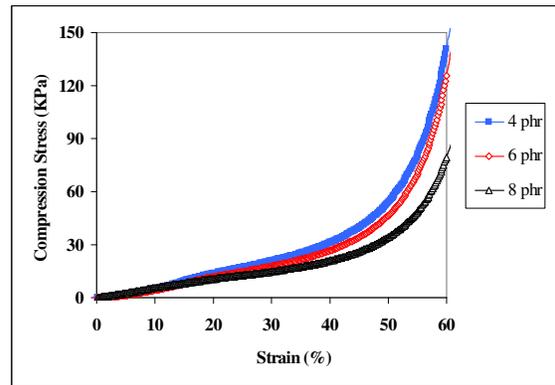


Figure 9: Compression stress for different amount of sodium bicarbonate

Beside that, increase in cell size which is parallel to the foaming direction will causes longer distance for the upper and lower cell wall to be in contact resulting higher maximum strain. A sudden drastic change in the compression stress value for all the foam is caused by the effect of compression stress on the matrix phase [10].

The Effect of Different Ratio of Accelerator between TMTD and CBS

Rheology

The investigations are continued using different amount of accelerator between TMTD and CBS. The combinations of the two accelerators are fixed with 3.5 phr and only the ratios are altered. Based on the rheograph generated, the cure characteristic of the three compounded rubber show similar trend with only a slight change when scrutinized closely and the result is shown in Figure 10 and 11.

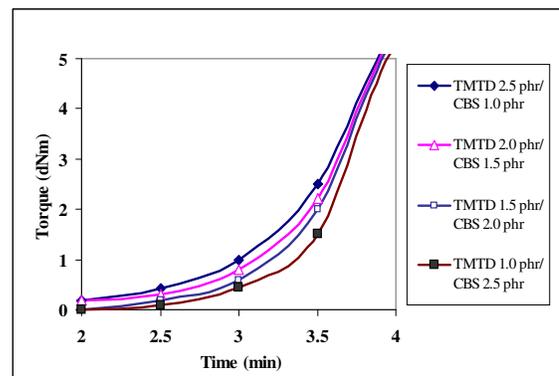


Figure 10: Rheograph for 2 until 4 minutes for 60 phr ENR-25 and 40 phr SMR-L with different ratio of accelerator between TMTD and CBS.

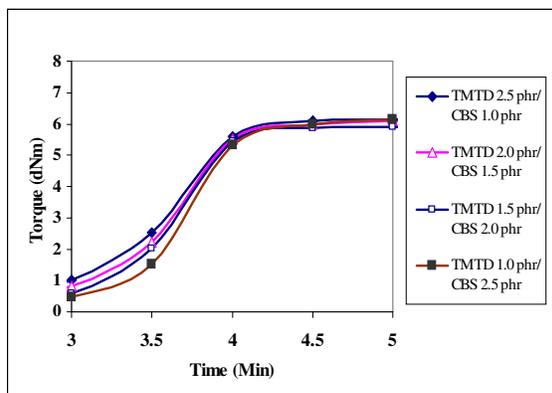


Figure 11: Rheograph for 3 until 5 minutes for 60 phr ENR-25 and 40 phr SMR-L with different ratio of accelerator between TMTD and CBS.

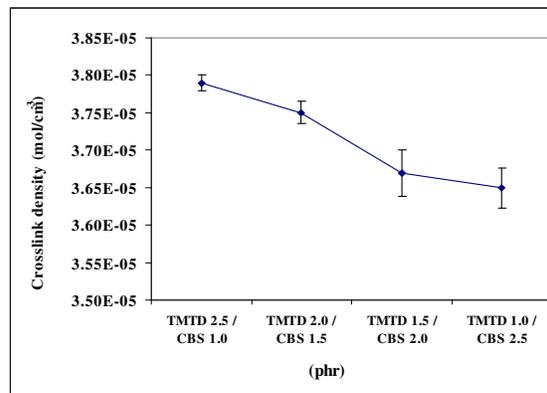


Figure 11: Crosslink density for different ratio of accelerator between TMTD and CBS.

The cure characteristic data obtained in table 8

Table 8
Cure characteristic data for different ratio of accelerator

Properties	TMTD 2.5 / CBS 1.0	TMTD 2.0 / CBS 1.5	TMTD 1.5 / CBS 2.0	TMTD 1.0 / CBS 2.5
M_L (dNm)	0.05	0.16	0.06	0.09
M_H (dNm)	6.26	6.21	6.15	6.13
Optimum cure torque, T_{90} (dNm)	5.57	5.54	5.53	5.50
Optimum cure time, t_{90} (min)	4.92	4.97	5.44	5.61
Scorj time, t_2 (min)	1.89	1.99	2.15	2.31
Minimum torque time, t_1 (min)	0.91	0.60	0.90	0.82

presented that the scorch time t_2 and the optimum cure time t_{90} are lengthened with adding higher ratio of CBS. Ismail & Hashim [11] claims that the function of CBS is to provide a secure processing stage by prolong the compound scorch time, While, the function of TMTD is to accelerate the formation of crosslinking. This is due to TMTD is a sulphur donor which contribute to increasing rate of crosslinking. According to Debapriya and colleagues [12], since sulphur exist in TMTD providing more space and place for the chemical crosslinking to begin which result in rising value of the crosslink density in the cured rubber matrix. This was proven experimentally by increasing ratio of CBS leads to lengthen the scorch time t_2 and the optimum cure time. More over, the maximum torque M_H also increase with additional higher ratio of TMTD which is supported by the crosslink density test show in Figure 11.

Physical Properties and Morphology of the Rubber Foam

The effect of time and rate of crosslinking play a major role in ensuring the foam stability and its physical properties. For elastomer foam, the purpose of crosslinking is to secure the foam from collapsing even though it is been expose under thermal degradation for a certain time. According to Park Chung [13], if the formation of crosslinking is insufficient, it will definitely make the cell damage or collapse. Figure 12 shows that as the ratio of CBS increase, the cell size parallel to the foaming direction seems to be enlarged in comparison to that of in the perpendicular direction of foaming.

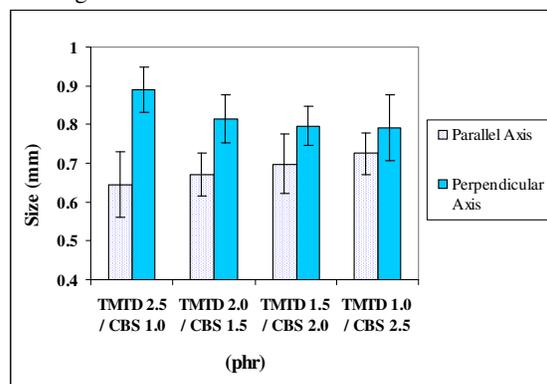


Figure 12: Cell size for different ratio of accelerator between TMTD and CBS.

This could be caused by the prolonged induction time resulting in reduction crosslinking formation in the matrix phase. Benning [14] claimed that the crosslinking must already formed before the decomposition of the blowing agent reach to an end because the melted rubber would flow and could not maintain the cell wall from collapsing.

The gas generated during the decomposition of the blowing agent will give an extensional force from within the matrix and the force will only remain for a short period of time because the gas then will diffused

through the walls thus collapsing the cell wall. With the cell compressed in place, the cell size parallel to the foaming direction will decreased, while the cell size perpendicular to the foaming direction will increased as shown in Figure 13.

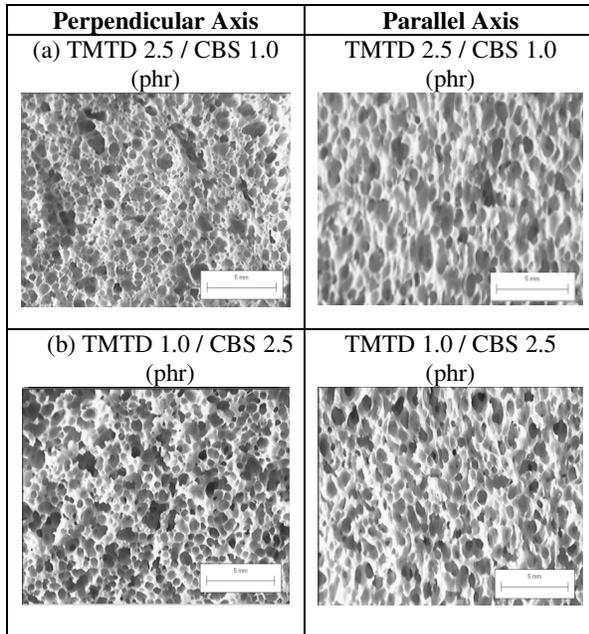


Figure 13: Cell distributions for different ratio of accelerator between TMTD and CBS.

Referring to Figure 14,

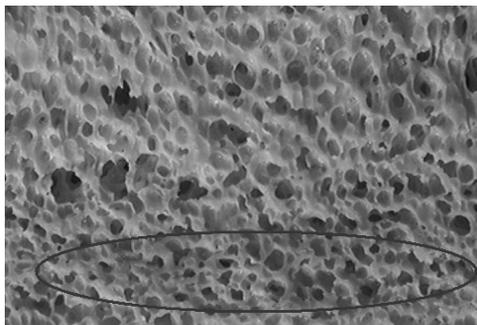


Figure 14: The cross sectional overview at the parallel side of the rubber foam with the ratio of accelerator of 2.5 phr TMTD and 1.0 phr CBS.

the cell collapse is much more obvious at the bottom part of the foam and this will affect the crosslink density and the ratio of foaming expansion. The cell morphology and the foam density are closely related to each other where as the cell size parallel to the foaming direction decreased, it will also caused the expansion ratio of the foam to drop as shown in Figure 15.

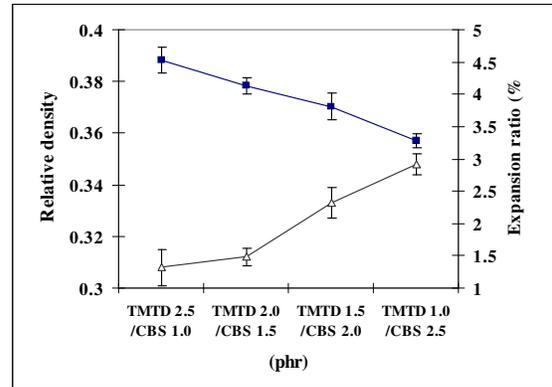


Figure 15: The effect of relative density and the ratio of foaming expansion for different ratio of accelerator between TMTD and CBS.

Mechanical properties of the rubber foam

For compression set, the value declined significantly to increasing amount of CBS. It can be clearly seen in Figure 16.

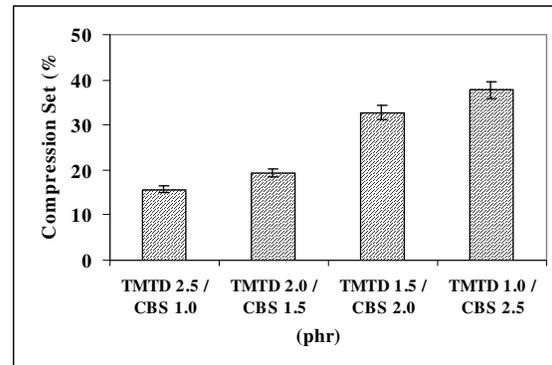


Figure 16: Compression set for different ratio of accelerator between TMTD and CBS.

These scenario is caused by the average cell size are getting bigger with increasing amount of CBS leading to microscopic deformation when the foam are exposed to heat and compression (6). As for compression stress test, bigger cells will undergo microscopic deformation sooner than the smaller cells in the non-linear region. However, Gibson and Ashby explained that foams with relative density of more than 0.3g/cm^3 is much likely to be influenced by the matrix phase. Due to this matter, the data for compression stress test rose to with increasing value of the foam density even though the average cell size are getting bigger with adding higher ratio of CBS as shown in Figure 17.

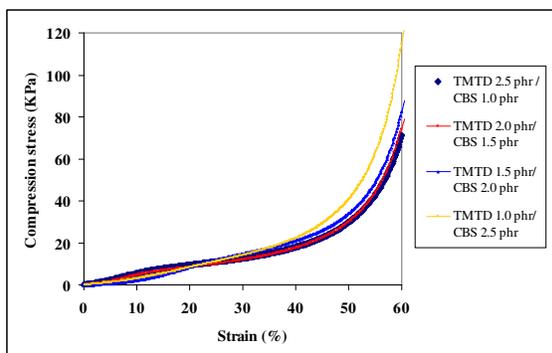


Figure 17: Compression stress for different ratio of accelerator between TMTD and CBS.

Since foam with the ratio of 1.0 phr TMTD already show that the cell collapse during curing, the foam itself shows deviation from the usual trend (refer Table 9).

Table 9

Data for compression stress for different ratio of TMTD and CBS

Rubber foam	TMTD 2.5 / CBS 1.0	TMTD 2.0 / CBS 1.5	TMTD 1.5 / CBS 2.0	TMTD 1.0 / CBS 2.5
Compression modulus (kPa)	0.545 ± 0.021	0.478 ± 0.010	0.698 ± 0.008	0.657 ± 0.018
Compression stress at 25% strain rate (kPa)	12.707 ± 0.018	11.687 ± 0.021	16.798 ± 0.014	15.598 ± 0.024
Compression stress at 50% strain rate (kPa)	29.241 ± 0.014	32.841 ± 0.009	37.587 ± 0.017	46.452 ± 0.017
Maximum strain (%)	69.134 ± 0.009	68.972 ± 0.014	67.854 ± 0.012	67.137 ± 0.012

Besides the increasing value of the non-elastic linear region, the maximum compression strain was also affected. With a rise in the density and compression stress value it is obvious that the strain that can be supported by the foam will reduce with increasing content of CBS. Again the formulation with the ratio of 1.0 phr TMTD show the highest value of compression stress at 50% of compression strain compare to other formulations. This was due to higher amount of matrix phase at the bottom of the foam which leads to a sudden increased value of compression stress at 60% of compression strain.

Conclusions

It is proven from the result of this study that using sodium bicarbonate alone is capable in exerting enough carbon dioxide to produce rubber foam. It is also discovered that formulations with higher amount of ENR-25 undergo shrinkage but the shrinkage seems to be diminished as seen in blend with 60 phr ENR-25 and

40 phr SMR-L. This blend ratio is able to produce stable rubber foam due to faster curing compared with 100 phr ENR-25 and 80 phr ENR-25 blend with 20 phr of SMR-L. Shorter the cure time will lead to stronger the cell wall which could prevent the expanded rubber from collapsing or shrinkage.

From the study conducted on the stable 60 phr ENR-25 and 40 phr SMR-L blend foam, it was found that lower amount of sodium bicarbonate will generate smaller cell size, thicker cell wall, lower value of expansion ratio, higher value of relative density and higher reading for compression stress. But the compression set test presents an increment trend with increasing cell diameter. This was contributed by smaller cells exist in between the cell wall which minimizes the microscopic collapsing and promote recovery to the rubber foam.

Results for study on the usage of different ratio of accelerator showed that the induction time is prolonged with increasing ratio of CBS. This is because CBS is not an effective sulphur donor compared to TMTD. The use of higher content of CBS results in bigger average cell size, increasing cell wall thickness, lower expansion ratio, higher relative density and increase value for compression set. For compression stress, the values are rising with higher ratio of CBS which due the fact that the produced foam are strongly influence by the matrix phase and subsequently caused the relative density to be higher.

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