AGEING OF SIDEWALL RUBBER COMPOUNDS

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AGEING OF SIDEWALL RUBBER COMPOUNDS

ABSTRACT

The ageing of sidewall rubber compound was studied under ambient temperature over seven weeks. Results showed significant increase in mooney viscosity, RPA (G' at 1%) and RPA (G' at 100%) over ageing time ($p < 0.005$). Aging sidewall compound for seven weeks did not cause any significant decrease in structural integrity in the rheometer properties, tensile, elongation, modulus, hardness, rebound and RPA (tanD at 10%). Longer time required to see the physical changes during ageing of sidewall compound. Thus, this study should be repeated using accelerated ageing process to see what is the significant changes in the physical properties during sidewall rubber compound ageing process.

Keywords: ageing, sidewall, rubber, compounds

PENUAAN KOMPOUN GETAH “SIDEWALL”

ABSTRAK

Penuaan kompoun getah ‘sidewall’ telah dikaji di bawah suhu bilik selama tujuh minggu. Keputusan menunjukkan peningkatan ketara dalam kelikatan mooney, RPA (G 'pada 1%) dan RPA (G' pada 100%) sepanjang masa eksperimen di jalankan ($p < 0.005$). Sepanjang proses eksperimen selama tujuh minggu, kompaun getah ‘sidewall tidak menunjukkan penurunan ketara dalam integriti struktur dari segi rheometer, tegangan, pemanjangan, modulus, kekerasan, pemulihan dan RPA (tanD pada 10%). Masa yang lebih lama diperlukan untuk melihat perubahan fizikal semasa penuaan kompoun getah “sidewall”. Oleh itu, kajian ini perlu diulang menggunakan proses penuaan dipercepatkan untuk melihat apakah perubahan ketara dalam sifat-sifat fizikal semasa proses penuaan getah “sidewall”.

Keywords: penuaan, sidewall, kompoun

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LIST OF SYMBOLS AND ABBREVIATIONS

ASTM	:	American Society for testing and Materials
BR	:	Butadiene rubber
LTDE	:	Long Term Durability Endurance Test
NHTSA	:	U.S. National Highway Traffic Safety Administration
NBR	:	Nitrile-butadiene rubber
P-END	:	Passenger Endurance test
RPA	:	Rubber Processing Analyser
SBR	:	Styrene-butadiene rubber

LIST OF APPENDICES

Part I - Mixing Details

Compound Name: Sidewall

Mixing room:

responsibility to fill in:

MIXING:

Observations:	Master Batch- OK	Master Batch- NOK	Master Batch-2 OK	Master Batch-2 NOK	Master Batch-3 OK	Master Batch-3 NOK	Final Mix OK	Final Mix NOK
load factor, ok?	☑	☐	☐	☐	☐	☐	☑	☐
ram position, ok?	☑	☐	☐	☐	☐	☐	☑	☐
Compound slippage?	☑	☐	☐	☐	☐	☐	☑	☐
Explosion during ram lifts?	☑	☐	☐	☐	☐	☐	☑	☐
cycle time, ok?	☑	☐	☐	☐ ☐	☐	☐	☑	☐
Tdp last batch	150						110	
Number of batches	8 Batches						5 Batches	

ADDITIONAL REMARKS (at least for NOK items):

DOWN-STREAM: **Observations:**	Master Batch- Yes	Master Batch- No	Master Batch-2 Yes	Master Batch-2 No	Master Batch-3 Yes	Master Batch-3 No	Final Mix Yes	Final Mix No
Undispersed material?	☐	☑	☐	☐	☐	☐	☐	☑
Sticking on mills/calander?	☐	☑	☐	☐	☐	☐	☐	☑
Sticking on conveyor?	☐	☑	☐	☐	☐	☐	☐	☑
Compound too dry?	☐	☑	☐	☐	☐	☐	☐	☑
Compound too soft?	☐	☑	☐	☐	☐	☐	☐	☑
Bypass on tandem used?	☐	☑	☐	☐ ☐	☐	☐	☐	☑
Rough or smooth surfac								

REMARKS:

For trial, mixing was run as normal without changed any parameters.

process capability - continuous production possible?: YES ☑ NO ☐

Raw Materials:

Is additional dosing equipment needed? YES ☐ NO ☑

If yes, what kind of equipment?

Verified:

Date: #######

Name: Hazizi Akmar Bin Mukhtar

Function in plant: Engineer Material - Mixing

Compounder:

Please supply

Mixing curves (Temp. Ram, Rotor) MB: ☑

Mixing curves (Temp. Ram, Rotor) FM: ☑

CHAPTER 1: INTRODUCTION

1.1 Tire Technology

1.1.1 Tire construction

There are many different types of tire constructions. The three main common designs are bias tires, belted-bias tires, and radial tires. The radial design is the most popular for automobiles. This is because the construction is different and users may benefit in term of responsiveness and efficiency

1.1.2 Function of Pneumatic tire

A pneumatic tire is a composite that consist of structure of compounded rubber, steel and fabric. It is fitted or attached to a rim and wheel to support a vehicle and its load and work as a cushion of compressed air which is contained within the tire. The pneumatic tire function as:

i. Supporting the vehicle load
ii. Transferring driving and braking forces to the road surface
iii. Generating lateral forces for cornering and vehicle handling. This is to control and guide the direction of travel
iv. Providing safety through durability, manoeuvrability, snow traction, wet and dry traction, and high speed performance
v. Providing dimensional stability by undertaking only insignificant change of size or shape upon inflation
vi. Offering economy through long tread life (wear resistance) and low rolling resistance (energy consumption)

In order to perform this, the tire needed enough rigidity to develop substantial forces in all directions. Besides, it required enough flexibility to be able to face the obstacles without having damage and along fatigue life in flexing from a particularly curved shell to a flat surface and back.

1.1.2.1 Inner liner

Inner liner is the first layer inside the tire. The purpose of this inner layer is to prevent air penetration or air loss from the tires.

1.1.2.2 Bead and Bead Chaffer

The beads of the tire form the contact point between tire and wheel. The beads are prepared form high tensile strength steel wires and are enclosed by a hardened rubber compound. This is to ensure an airtight seal between the tire and wheel. The bead chafers rest between the bead and the body ply of the tires. This is to prevent the bead wires from damaging the tire casing. Besides, to improve the tire's handling by making the sidewall above more responsive.

Figure 1.1: Tires Bead

1.1.2.3 Sidewall

A tire's sidewall has many different and important functions. The function of sidewall of the tire is to provide protection for the inner layers from abrasions and cuts. Tire manufacturers are able to alter the handling characteristics and load carrying capabilities of the tire by changing the sidewall construction. For example, a stiffer sidewall may lead to more expectable handling but have to compromise the ride quality. The other function of sidewall is also as an identification for a tire where it is the location of important tire information such as maximum air pressure, size, load and speed rating (refer figure 1.4)

1.1.2.4 Ply

The ply is one of the most essential layers of the tire casing. It contains rubber-bonded cords that run through the circumference of the tire at 90 degrees from the direction of tire travel. The radial construction of the ply benefits in absorbing bumps on the road or driving surface. Beside it allows the sidewall and the thread to operate independently. This reduces flex in the tread, transverse slip and friction, which leads to more responsive handling, increased fuel efficiency, and longer tread life.

1.1.2.5 Belt Package

The tire's belt package plays multiple functions including preventing the tire casing from road damage and forming the flat area for the tire tread. The belt package is built from woven strands of high strength steel fibres. They are bonded to the rubber. The belt package increases tire rigidity and at the same time remaining flexible enough to absorb bumps in the road.

1.1.2.6 Cap Ply

Cap plies are important in performance tires and higher quality all seasons tires. The cap ply fights centrifugal forces. Besides, it contains the belt package by maintaining the tire shape at high speeds. Thus, it leads to a better handling and braking.

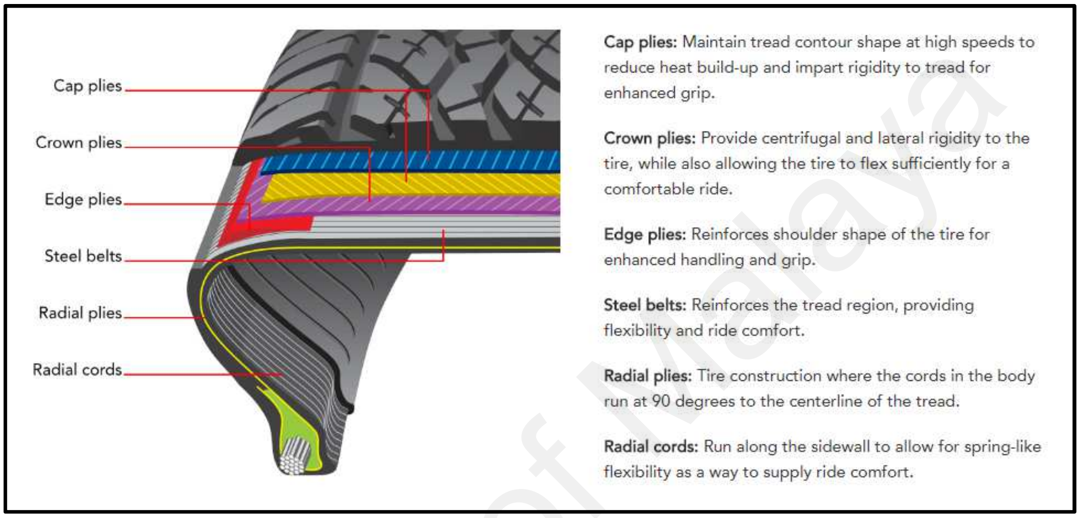

Figure 1.2 Tires Belt Package

1.1.2.7 Tread

The tread area consists of many different features. It works together to provide handling, traction and ride characteristics. These features will vary depending on the proposed use of the tire. Circumferential grooves are channels that run the full length of the tire. The function is to reduce hydroplaning by evacuating water away from the tread surface. By allowing air to pass under the tire, they also help reduce tire noise. Lateral grooves are channels that run 90 degrees from the direction of travel. They enhance traction by removing mud, water, and snow from the tread design.

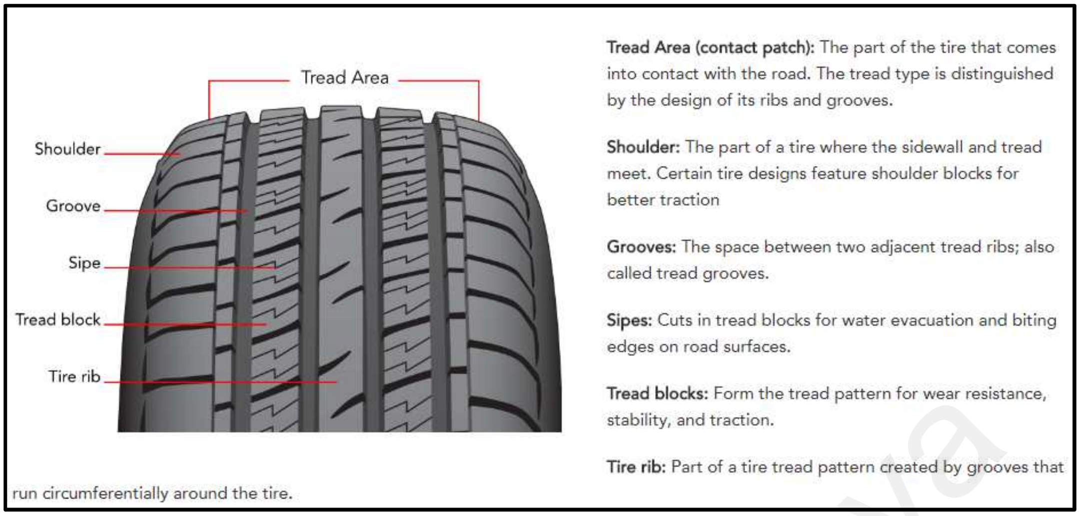

Figure 1.3 Tires Tread

1.2 Tire Compounding

Radial, Commercial Vehicle Tires (CVT), Off The Road (OTR) Tires are products of complex engineering. They are made up of numerous different rubber compounds, many different types of carbon black, fillers like clay and silica, and chemicals & minerals added to allow or accelerate vulcanization. The tires also have several types of fabric for reinforcement and several kinds and sizes of steel. Some of the steel is twisted or braided into strong cables.

1.2.1 Composition of Tires

Table 1.1 Tires Composition

Ingredient	**Passenger Car Radial (PCR)**	**Commercial Vehicle Tires (CVT)**	**Off The Road (OTR)**
Polymer	47%	45%	47%
Carbon Black	21.50%	22%	22%
Wire Metal	16.50%	25%	12%
Fabric Textile	6%	Nil	10%
Zinc Oxide	1%	2%	2%
Sulphur	1%	1%	1%
Additives	8%	5%	6%
Carbon-based materials	74%	67%	76%

As shown in table 1.1 tires contained so many different ingredients and compounds. This engineering miracles, are expected to handle the tortures of heat and cold, abrasive conditions, high speed and often inadequate air pressure. They are expected to perform for tens of thousands of kilometres and at the same time retain their essential properties despite poor driving habits and sometimes inadequately maintained or built roads.

1.2.2 Side wall tire compounding

The sidewall is an all-rubber component extruded into a specific profile. It is compounded to provide resistance to ozone and weather effects. Besides, it is also to provide resistance to abrasion. Overall it serves to protect the body plies. To serve the above functions, when subjected to severe distortion over a range of temperature conditions, the compound expected to maintain flexibility without cracking.

1.2.3 Component of sidewall rubber compound

To protect the casing from ozone attack, cracking, weathering and abrasion, typically, a sidewall rubber compound contains a blend of natural rubber and butadiene rubber with carbon black and many other chemicals. As for this experiment, the exact sidewall compound preparation will be discussed in chapter 3 (Methodology)

1.3 Tire ageing

1.3.1 Definition tire aging

Tire aging is an occurrence involving the degradation of the material properties of a tire overtime ('TIRE AGING : A Summary of NHTSA ' s Work', n.d.). The effect of tire aging it can compromise its integrity of its physical structures and risk its performance. Consequence of the aged tires, they are more prone to tire failure and spectrum of problems include tire cracking (figure 1.4). At best it may cause an inconvenience, or at worst may lead to a motor vehicle crash.

1.3.2 Mechanism of rubber compound aging

Rubber compound aging in the field is a thermo-oxidative process. The tire material properties degrade with increasing time and environmental factor like heat increase the speed of the degradation process. Figure 1.4 showed sidewall tire cracking as a result of tire ageing.

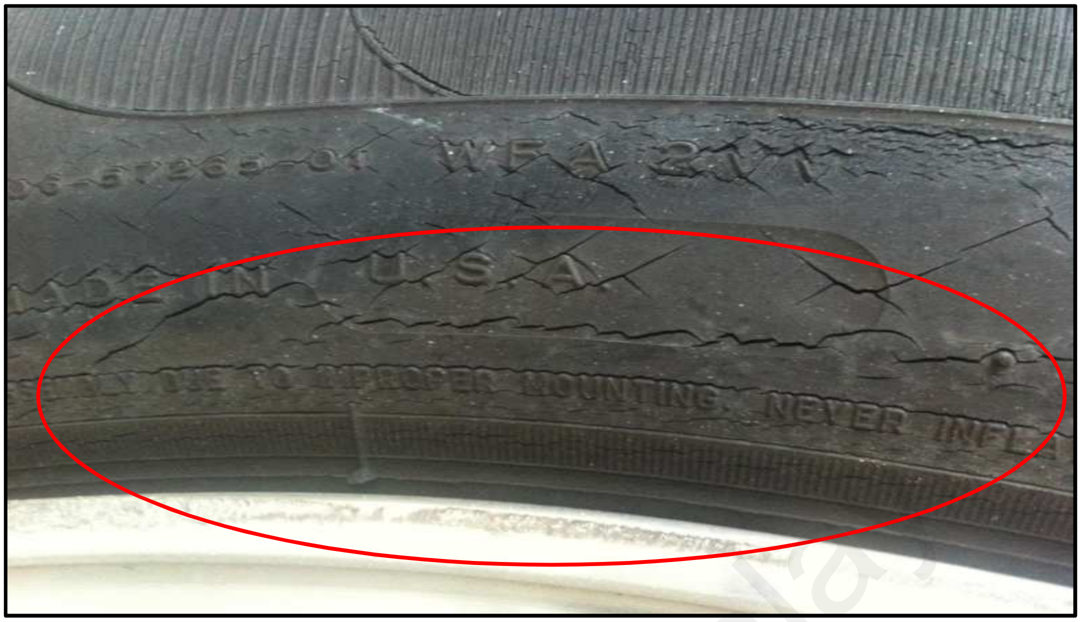

Figure 1.4 Sidewall Cracking on Tires

1.4 Problem statement

Evaluation of the long term performance of sidewall tire compound is very important. Currently the standard tests used by industry to evaluate the long-term performance of sidewall rubber compounds involve only one or at most two influencing factors. However, in real industry, the sidewall tire rubber compounds are subject to aging by a combination of factors including heat, ozone, oxygen, dynamic strain, flexing, ultraviolet light, and liquids. The effect of the interactions between these factors on aging is, essentially, unknown.

To our knowledge there was no previous study investigate on ageing properties behaviour of sidewall rubber compound. The investigation is essential to accurately predict the aging resistance of a sidewall compound in real industry.

1.5 Rationale of the study

This study to contribute in term of method to sustain long term performance of sidewall rubber compound

1.6 Research Questions

1. What are physical changes during sidewall compound ageing?
2. Is there any correlation between physical changes of sidewall compound over ageing time?

1.7 Study Objectives

The objective of this study:

1) To describe physical properties changes during sidewall rubber compound ageing and
2) To determine the correlation between physical changes of sidewall rubber compound and ageing time

CHAPTER 2: LITERATURE REVIEW

2.1 Tire aging methods evaluation

There were two ways of evaluating tire aging. Either to test the real tire aging during field study or using accelerated method to mimic the real field study.

2.1.1 Tire aging field study

In the spring of 2003, NHTSA (Hs, 2014) conducted a tire aging field study involved comparing in service tires (up to 7 years old tire), spare tires (up to 10 years old) and compared to new tire of the same model and determine the rate of degradation in tire performance and physical properties. Several test were performed include peel strength and tensile strength test. The study found a reduction in peel (adhesion) strength between the steel belts, an increase in hardness of most rubber components, a loss of the rubber components' ability to stretch, increased crack growth rates, and a reduction in cycles to failure in fatigue tests

2.1.2 Accelerated tire aging study

a) **Long Term Durability Endurance Test (LTDE) and Passenger Endurance (P-END) test**:

LTDE and P-END test is a combined tire aging and durability test. In this test, the tire is inflated using an oxygen-enriched air mixture and run on an indoor road wheel for up to 500 hours and 240 hours respectively at elevated loads and pressures to fatigue the tire structure and induce heat, which in conjunction with the oxygen-enriched inflation mixture accelerated the aging process (Hs, 2014). The study found these methods tend to cause "over aging" some parts of the tire which was not consistent with real field study.

b) **Oven-Aging Method**:

The tire is inflated using the same oxygen-enriched air mixture previously mentioned, and heated in an oven for a period of time to accelerate the aging process by speeding up chemical reactions and material property changes (Hs, 2014). The study found the Oven-Aging method was the only method successful at replicating the overall material properties and stepped-up load road wheel results of the used tires.

2.1.3 Physical properties changes during tire ageing

Study by NHTSA (Hs, 2014) measured physical properties changes during tire aging. The results showed particularly, the hardness, modulus, oxygen content and cross-link density showed increasing in trend. In contrast, the tensile, elongation, peel adhesion, and flex properties tended to decrease over time. All of these changes are coherent with the suggested mechanism of thermo-oxidative aging.

Jie liu et al (Liu, Li, Xu, & Zhang, 2016) studied on Investigation of Aging Behaviour and Mechanism of Nitrile-butadiene rubber (NBR). The study evaluated mechanical properties using elongation and tensile test at room temperature (23° C). They found that the elongation at break decreased with increasing aging time. In contrast, the tensile strength increased in the first phase of the study and decreased after 70 days of thermal aging.

Kataoka et al (Kataoka, 2003) studied on Effects of Storage and Service on Tire Performance found the Shore A hardness of the tread rubber on a specific spare tyre was observed to increase by 10 over a period of approximately 250 weeks.

2.2 Aging behaviour of properties on NR/BR polymers blending

Blending of rubber widely used in tyre industries to enhance mechanical properties in tyre rubber compounding especially in elastic stability. However, exposing the compound to sunlight and ageing over a time, the rubber compound hardens thus, as a result the tensile properties of the rubber material and the behaviour of the strain-energy density function are changed, greatly reducing the performance of the rubber product (Byungmoon et al 2018). In other way around, Natural rubber (NR) undergoes chemical changes on heat and air ageing. These changes affect its physical properties and as such, affect the service life of the rubber compound.

Study by Ahagon et al (1990), a vulcanized NR compound of a typical engine mount composition was subjected to thermo-oxidative ageing at temperatures from 70 to 110°C, to assess the effect on the tensile properties. The kinetics of degradation of the rubber compound, in terms of changes in these properties, was investigated. A fractional rate law was used to describe the kinetics of ageing in terms of its effect on modulus. Rates of ageing, in terms of effect on modulus, passed through a minimum at about 80°C, indicating the danger of trying to extrapolate in-service ageing behavior from high temperature ageing data. The activation energy of ageing in terms of its effect on modulus, determined for temperatures of 90–110°C, was 151 kJ mol^{-1}. A second order rate law was used to describe the kinetics of ageing in terms of its effect on tensile strength and elongation at break, with activation energies of 88.32 and 74.3 kJ mol^{-1}, respectively.

2.3 Thermal ageing effect on mechanical properties

The focus of this research was to investigate the effect of thermal degradation upon the mechanical properties of a natural rubber compound. Scott W. Case et al (2003) demonstrated the examined for both the quasi-static and dynamic mechanical properties of a natural rubber vulcanization which had been subjected to isothermal, anaerobic aging. The thermal aging was conducted between the temperatures of 80 °C and 120 °C for times ranging from 3 to 24 days. The effect of thermal degradation was measured using the changes in the crosslink distribution of the vulcanizes as functions of time at temperature. A master curve relationship between the crosslink distribution of the vulcanizes due to thermal degradation and the static and dynamic mechanical properties has been developed. It was found that the both the quasi-static and dynamic mechanical properties correlated with the percentage of poly and monosulfidic crosslinks, where in general higher levels of polysulfidic crosslink gave rise to the highest mechanical properties of rubber compound.

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CHAPTER 3: METHODOLOGY

3.1 Study Design

This is an experimental study.

3.2 Materials and compound preparations

The sample of sidewall rubber compound were tested based on recipe in table below:

Table 3.1: Sidewall rubber compound recipe

Material	PHR
Standard Malaysia Rubber (SMR 20)	34.5
Synthetic Rubber BR	54
SBR 1500	11.5
Reclaim Rubber	20
Carbon Black N339	43
Aromatic Oil	1
Phenolic Resin	2.1
Antioxidant DTPD	1.1
Ozone Protect Wax	2.25
Zinc Oxide	2.9
Accelerator	0.85
Soluble Sulphur	1.4

3.2.1 Mixing preparations

In this experiment, mixing process was conducted using tangential mixer that consists of 4 rotor wings and 270 Litre of capacity in volume. Optimization in batch weight is very important to produce better dispersion of rubber to interact with filler and chemical. During this experiment, at first stage of mixing, all chemicals except sulphur and accelerator was mixed together with SMR20, SBR, and NR to provide a well-mix mixture before proceed with compounding. Figure 3.1 showed the mixing curve for this experiment.

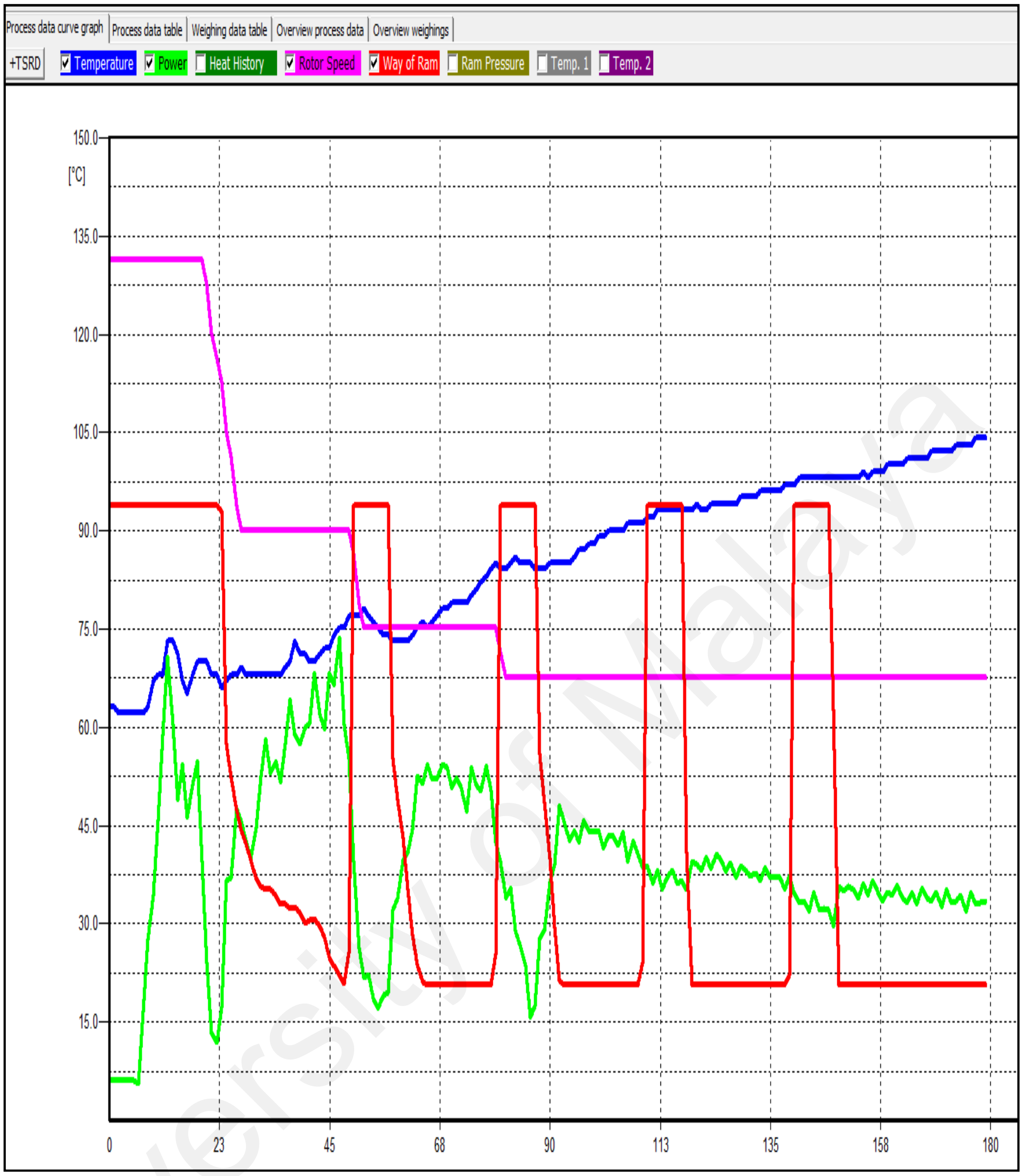
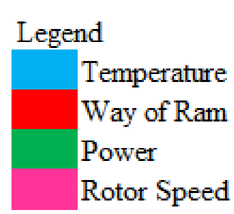


Figure 3.1: Mixing Curve



3.2.2 Sample preparation

Sufficient size and thickness of sample shall take into account to avoid any variables occurred during curing and testing processes. Besides, relaxing time after curing process is essential prior to physical testing to avoid any influences such temperature and dirt that may affect the final result. For the purpose of this experiment, the sample was prepared based on guideline ASTM. The samples used for the experiment were 6mm to 9mm thick to get ideal time and temperature during curing process. Besides, 16 hours was used for relaxing time after curing.

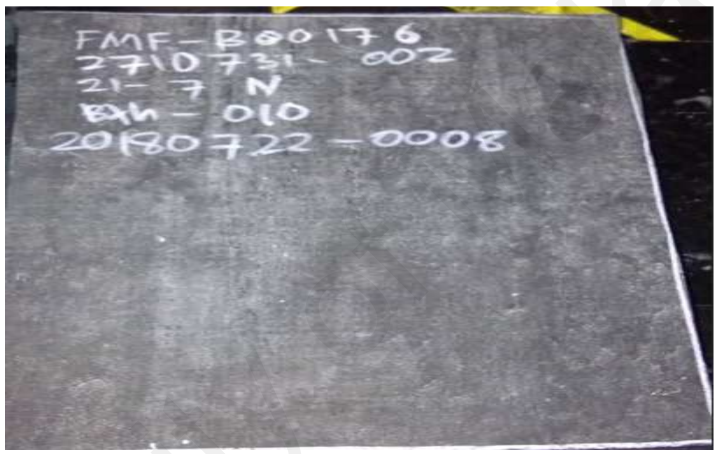


Figure 3.2: Raw Sample of Rubber Compound

Figure 3.3 Sample Thickness in Range (6mm to 9mm)

3.3 Ageing testing method

The rubber sidewall compound underwent normal ageing process at ambient temperature for seven weeks. Temperature and humidity were recorded weekly. The samples were cut in sufficient size and were used for the physical test. Results were recorded and analysed to see any differences to the physical properties during the ageing time. The main aim was to see the degradation of material properties as a function over aging time.

3.4 Physical testing

In rubber industry, physical testing is very important to measure the properties of rubber after cured. Even though a typical rubber product probably will never be stretched anywhere close to its ultimate for example in tensile result, many rubber product users still consider it as an important indicator of the overall quality of the compound. Therefore, physical properties of rubber compound were very important to compounder to ensure the results met the specification.

3.4.1 Rheological Properties MDR160

The rheological properties testing is very important in aspect of compounding process ability.

3.4.1.1 Scorch Time

The time to scorch is the time required at a specific temperature or heat history for a rubber compound to form incipient crosslinks. When a scorch point is reached after a compound is exposed to a heat history from processing side, the compound cannot be processed further to next processing. Therefore, scorch measurement is very important in determining whether a given rubber compound can be processed in a particular operation.

3.4.1.2 Cure Rate

Cure rate is the speed at which a rubber compound increases in modulus at a specific cure temperature or heat history. Cure time refers to the amount of time required to reach specific states of cure at a specific cure temperature or heat history. An example of cure time is the time required for a given compound to reach 50% or 90% of the ultimate state of cure at a given temperature.

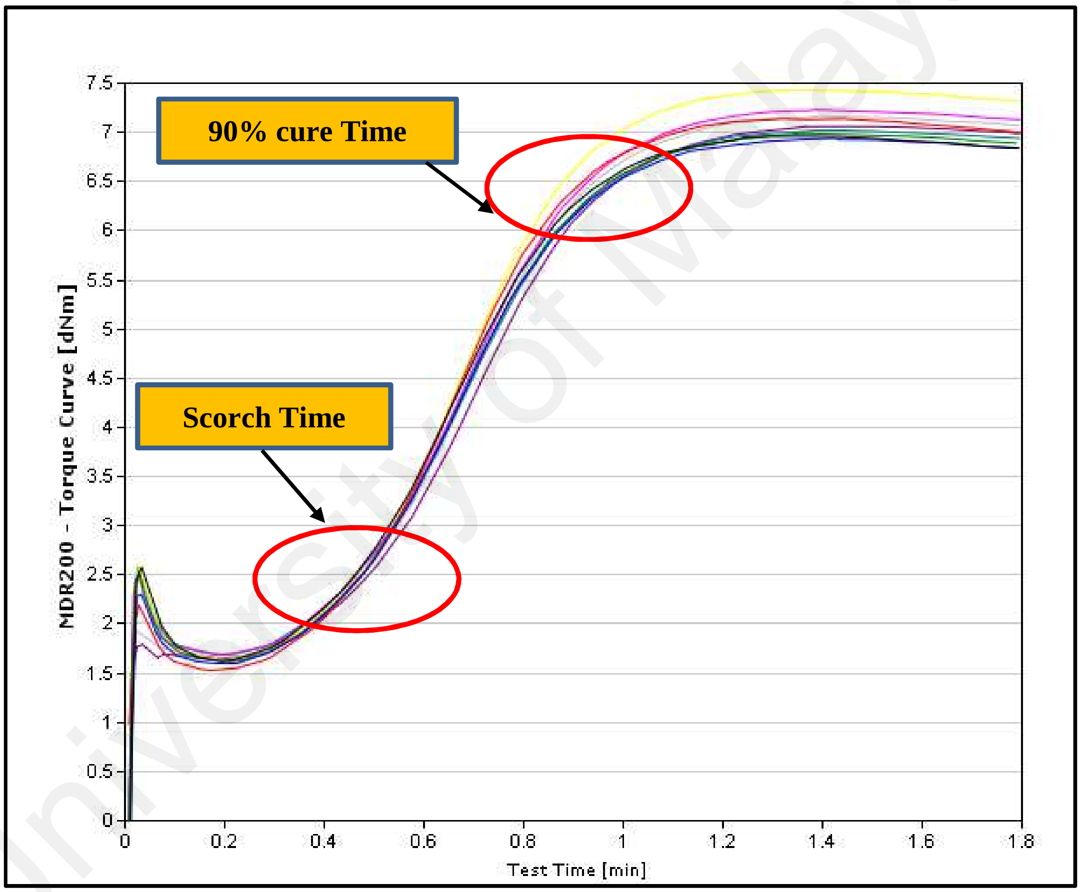


Figure 3.4 Sample of Rheological Curve

3.4.2 Mooney Viscosity

Viscosity is the resistance of a rubber, to flow under stress. Mathematically, viscosity (η) is shear stress divided by shear rate as equation (3.1) shown below,

Equation (3.1)

$$\eta = \frac{Shear\ Stress}{Shear\ Rate}$$

The mooney viscosity was performed using mooney viscometer machine with a one minute pre heat and run for three minutes. The lowest viscosity value in the last 30 seconds of the test was taken as the end result

Figure 3.5 Mooney Viscometer Machine (Source: Alpha Technology)

3.4.3 Tensile and Elongation

A dumbbell- shaped and ring test were used to measure the tensile and elongation of the rubber compound respectively. Tensile strength is calculated by dividing the load at break by the original minimum cross-sectional area. The result is expressed in Megapascals (MPa) and reported to three significant figures whereas percent elongation was calculated by dividing the elongation at the moment of rupture by the initial gauge length and multiplying by 100.

Equation (3.2)

$$Tensile\ (MPa) = \frac{Load\ at\ break}{Original\ width\ X\ Original\ Thickness}$$

Equation (3.3)

$$Elongation\ (\%) = \frac{Elongation\ at\ Rupture\ X\ 100}{Initial\ Length}$$

Figure 3.6 Gripping Samples

3.4.4 Modulus and Hardness

Modulus is the measure of the stiffness of a material. It measured stress at a specified elongation determined from a stress-strain using a tensile tester. As for modulus, the stress was measured at 300% using tensile tester. Hardness measurement is based on the depth measurement of a spike penetration with defined dimensions into material. Hardness also been measured to determine reversible deformation of the rubber compound.

Figure 3.7 Hardness Rubber Machine

3.4.5 Rebound

Rebound resilience is the ratio of the energy of indenture after impact to its energy before impact expressed as a percentage. Rebound is determined by calculating the height of the rebound of the standard needle when it is dropped from a certain height on the surface of the rubber material which is kept for the test.

3.4.6 Rubber Processing Analyser (RPA)

The purpose of Rubber processing analyser was to analyses the behaviour of rubber compound before and after cure. The RPA data was used to determine about the process ability, cure characteristic and cure speed.

Figure 3.8 Rubber Process Analyser (RPA) Machine (source: Alpha Technology)

3.4.7 Data Collection and Data Analysis

The measurement of physical properties reading was recorded weekly for seven weeks. The graphs were plotted. Spearman Correlations (correlation methods to measure ordinal/ continuous data) was used to measure the correlation of each measured property to duration of test. This correlation only determines if there is a significant changes and the direction of that change, but do not provides information about the magnitude of the change.

CHAPTER 4: RESULTS

4.1 Results of Physical properties

This experiment was to investigate if there are significant changes in any measured property correlated to the conditions of aging. The correlation value ranges between -1 for perfect inverse correlation to +1 for direct correlation. Zero value representing no correlation. In this experiment, the terms in Table 4.1 will be used as general descriptions of the level of correlation (Hs, 2014):

Table 4.1: Term of Strength of Correlation Used

Strength of Correlation	Correlation range
Insignificant	-0.39 to 0.39
Weak	-0.59 to -0.40 | 0.40 to 0.59
Moderate	-0.79 to -0.60 | 0.60 to 0.79
Strong	-1.00 to -0.80 | 0.80 to 1.00

4.1.1 Rheological Properties MDR160

According to the formula and recipe given in chapter 3, based on calculation, target and range of rheological properties has been set according to amount of materials in the compound. Figure 4.1, 4.2 and 4.3 showed the result for MDR160 for T10, T40 and T90 throughout the seven weeks of ageing time. The Spearman correlation for MDR160 for T10, T40 and T90 was 0.3929, 0.3784 and 0.5946 respectively indicate weak correlations. The correlations were not statistically significant as the p value > 0.005

Legend
Target
Range

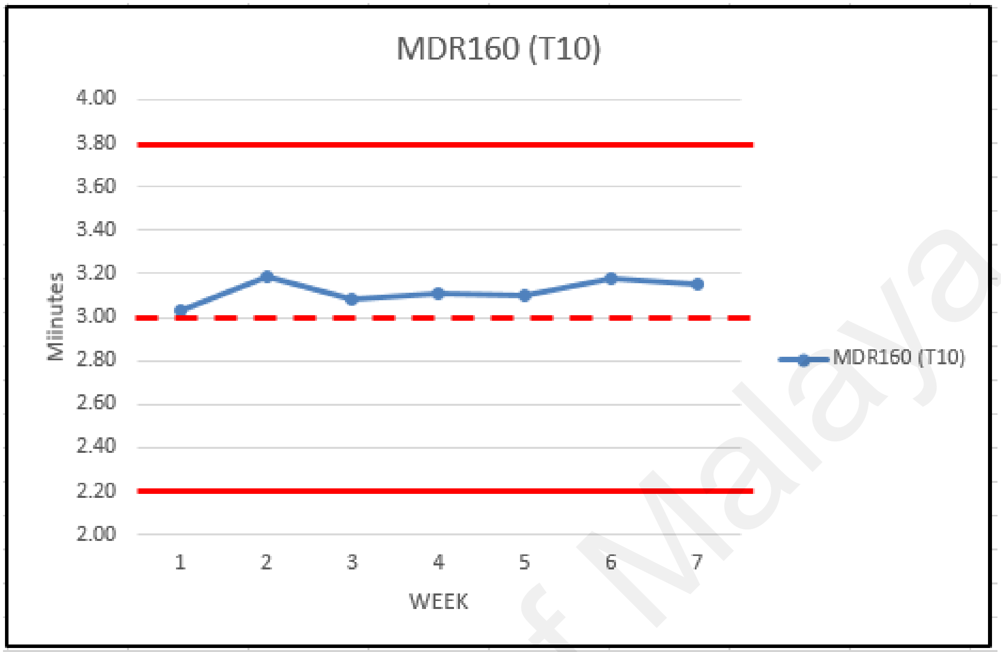


Figure 4.1 Result T10 MDR160

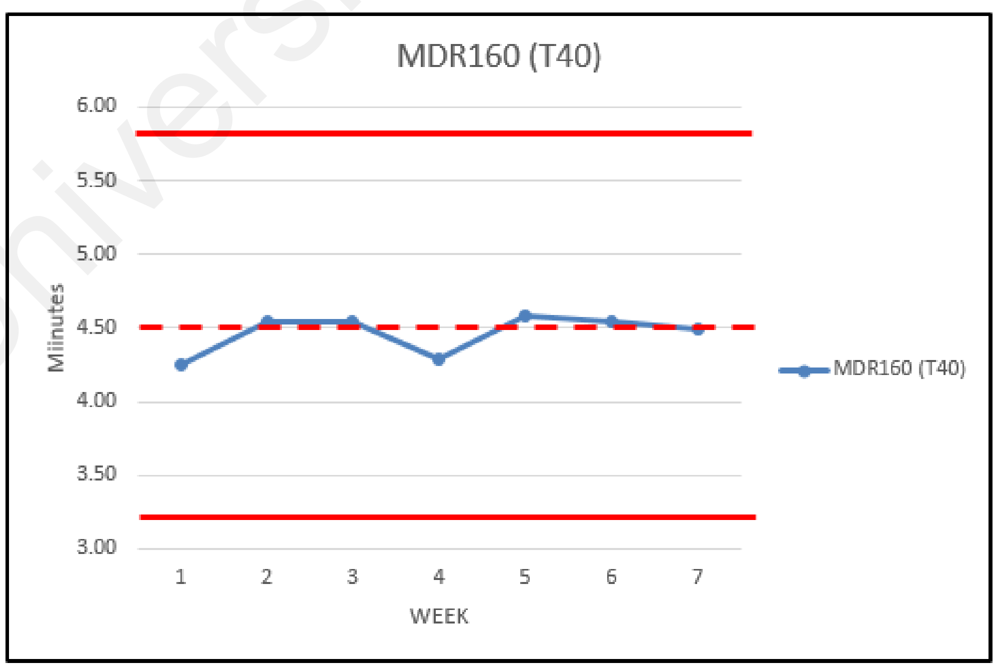


Figure 4.2 Result T40 MDR160

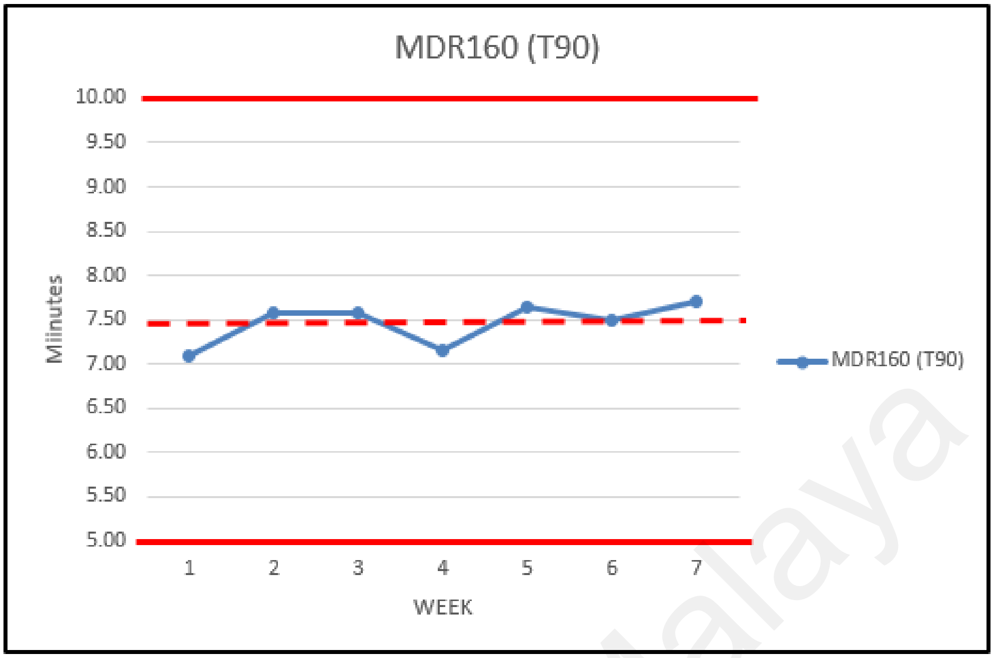


Figure 4.3 Result T90 MDR160

4.1.2 Mooney Viscosity

Theoretically, the measured of viscosity of rubber compound increase with running of time. However, it depends on the types of rubber compound and testing parameters that were used.

Figure 4.4 showed mooney viscosity increased with increased of the ageing time. After week six the mooney viscosity exceed the normal range. This can be due to environment storage temperature and high usage of SMR20. There was strong correlation between mooney and ageing time as the result of Spearman correlation was 0.9643. The correlation was statistically significant with p value <0.005.

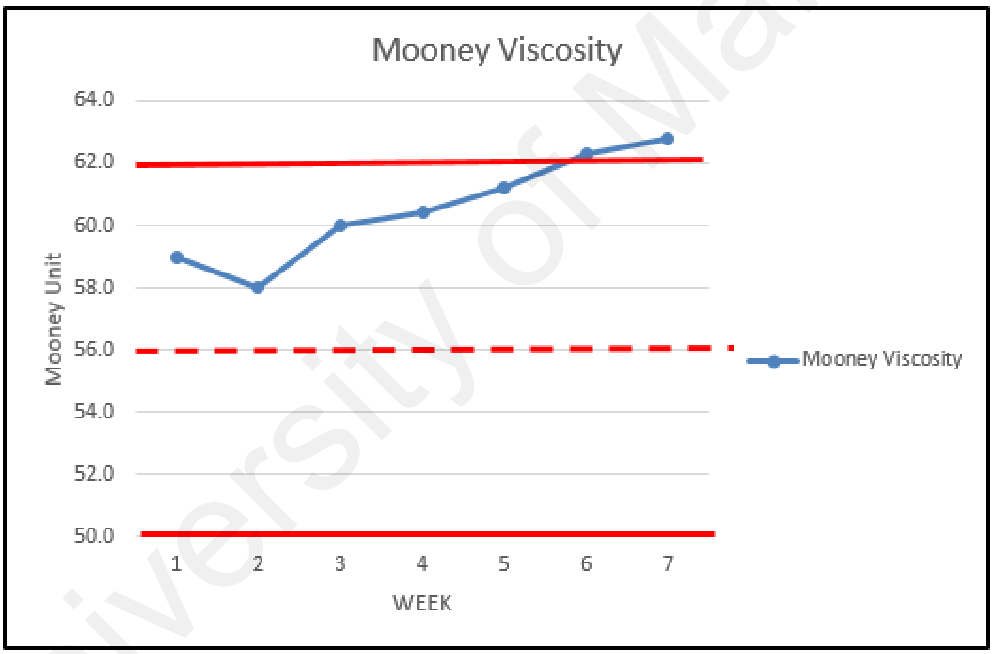


Figure 4.4 Result of Mooney Viscosity

4.1.3 Tensile and Elongation

Figure 4.5 showed the tensile strength of the compound throughout the seven weeks of ageing time. Noted the tensile strength decreased over ageing time. There was negative insignificant correlation of tensile over ageing time with spearman correlation value of -0.2143. The result was not statistically significant as p value was >0.005

Showing the same trend, Figure 4.6 showed reduction in the percentage of elongation throughout the seven weeks of ageing time. There was negative insignificant correlation of elongation over ageing time with spearman correlation value of -0.2143. The result was not statistically significant as p value was >0.005

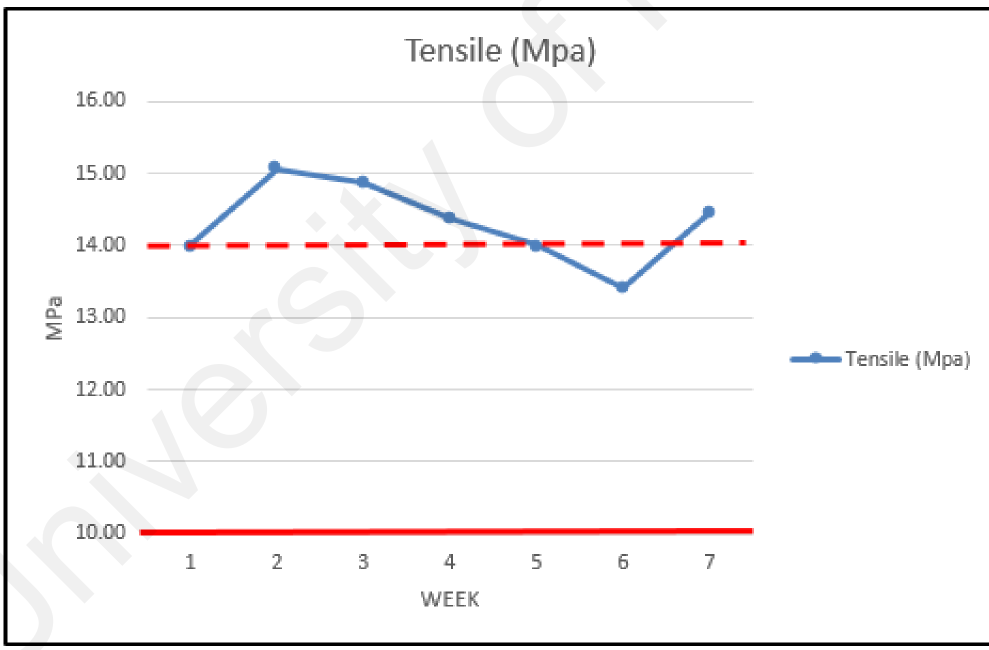


Figure 4.5 Tensile result

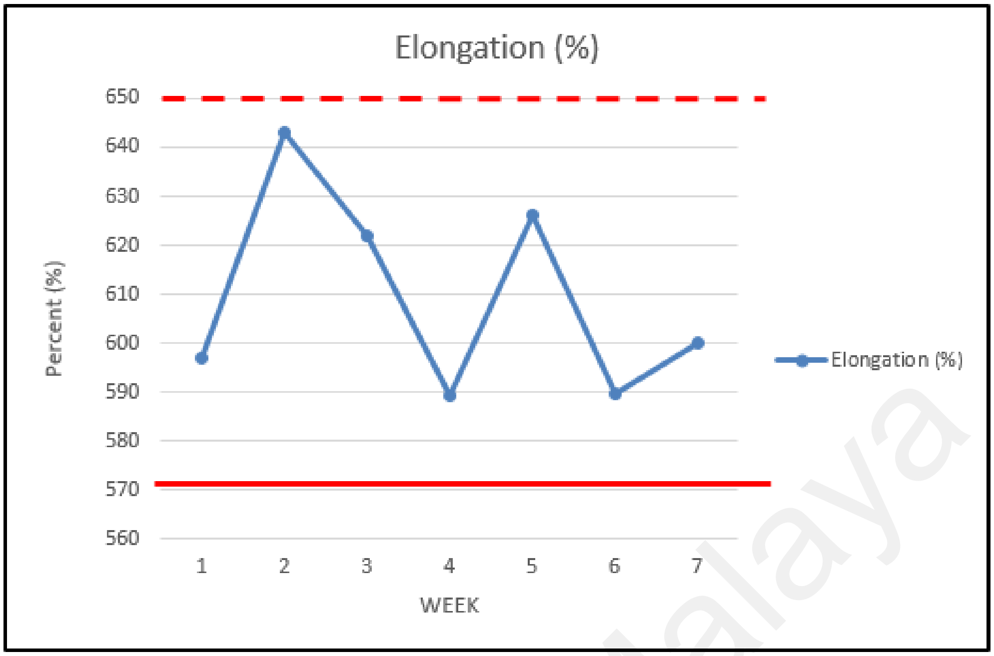


Figure 4.6 Elongation Result

4.1.4 Modulus and Hardness

Figure 4.7 showed the modulus result at 300% strain. Noted there was not much differences in the value of modulus throughout the ageing time. The spearman correlation was -0.071 indicate insignificant negative correlation with p value >0.005.

Meanwhile, Figure 4.8 showed the hardness result throughout the seven weeks of ageing time. Noted there was increased in the hardness as the time increased. The spearman correlation was 0.5766 indicate weak positive correlation. The result was not statistically difference with p value >0.005.

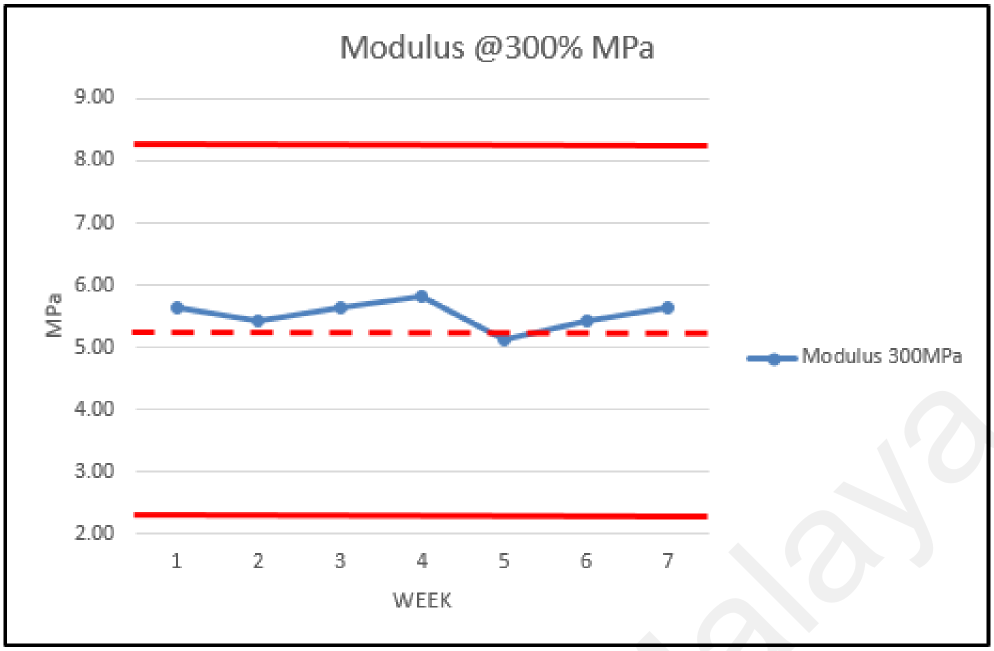


Figure 4.7 Modulus @300% result

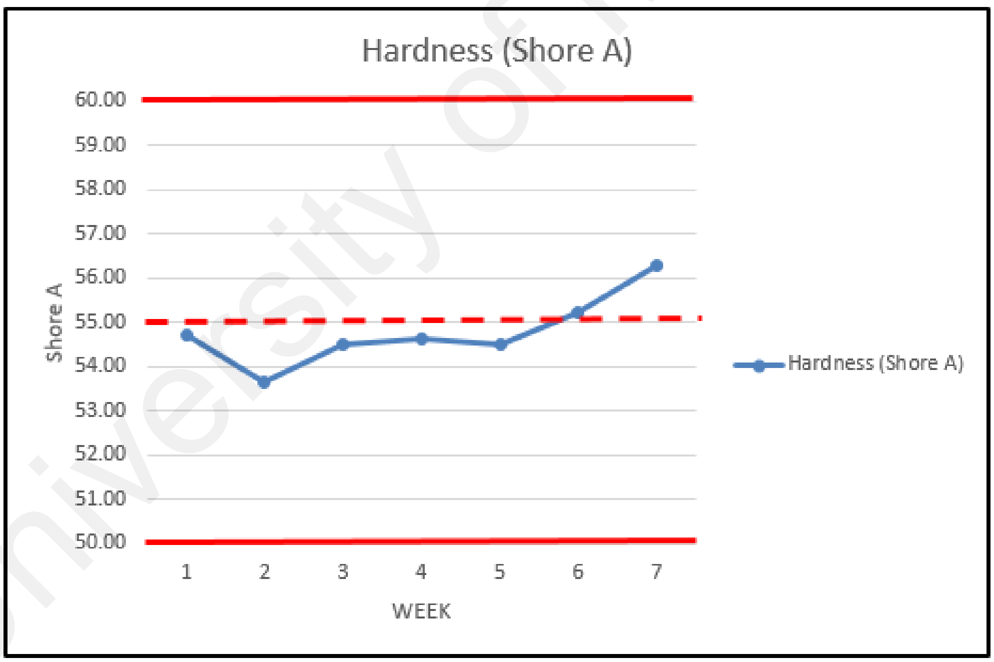


Figure 4.8 Hardness result

4.1.5 Rebound

Figure 4.9 showed the percentage of rebound throughout the seven weeks of ageing time. Noted there the spearman correlation was 0.1071 indicate insignificant correlation with p value >0.005.

Remaining damping or viscous quality of the cured rubber compound will reduce its rebound quality and increase its hysteresis or heat build-up quality from repeated deformations. Usually the higher the rebound of a compound is, the lower the hysteresis will be. However, this inverse correlation may not always be occurred, depending on different temperatures or different rates or amplitudes of deformation.

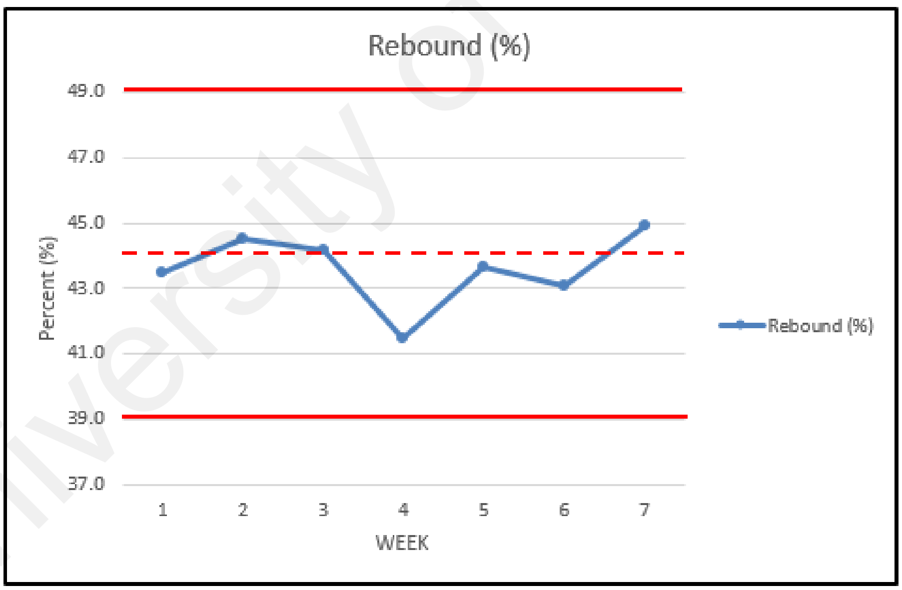


Figure 4.9 Rebound result

4.1.6 Rubber Processing Analyser (RPA)

RPA is very important in the analysis of behaviour of rubber compound before and after cure. The RPA data is used to determine the process ability, cure characteristic and cure speed. Figure 4.10, 4.11 and 4.12 showed RPA throughout the ageing time.

The spearman correlation for RPA (G' at 1%) and RPA (G' at 100%) was 0.7857 and 0.7500 respectively indicate moderate correlation. The results were statistically significant with p value <0.005. The spearman correlation for RPA (tanD at 10%) otherwise was -0.2000 indicate insignificant negative correlation with p value >0.005.

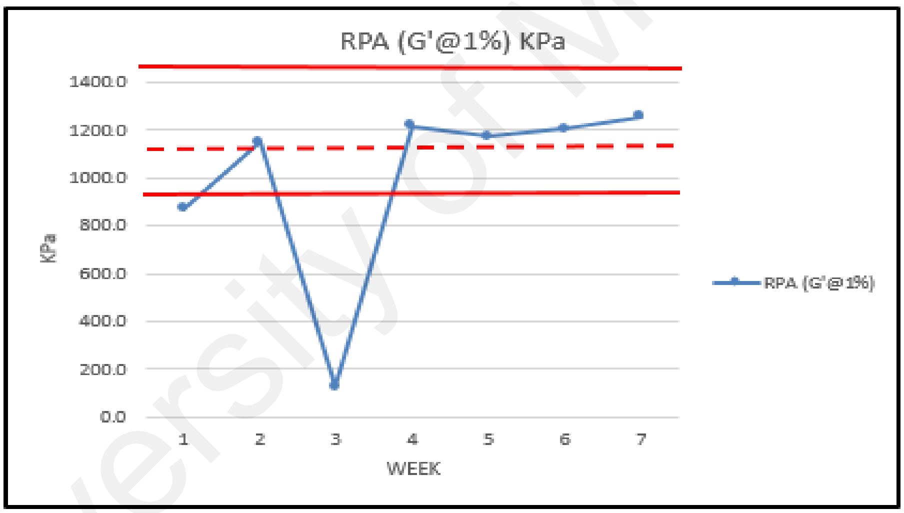


Figure 4.10 RPA (G' at 1%) result

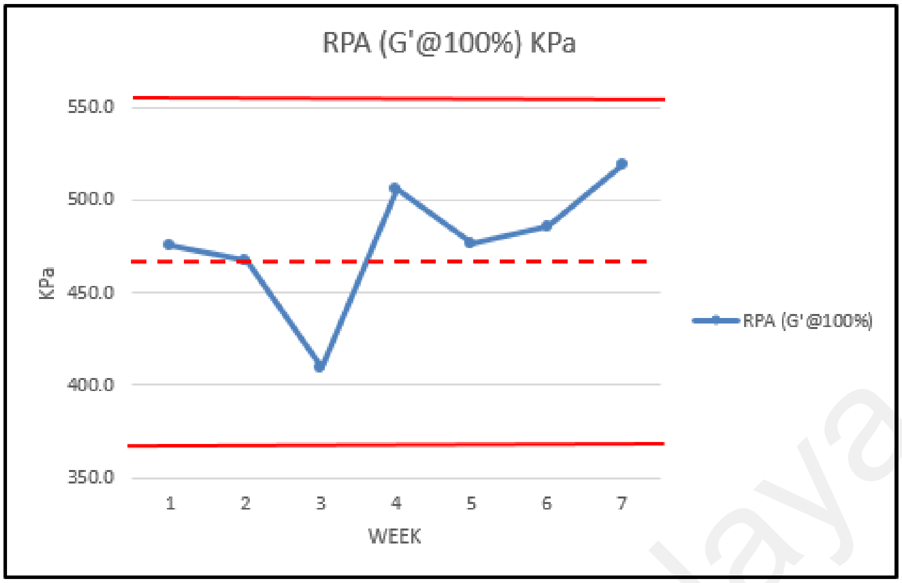


Figure 4.11 RPA (G” at 100%) result

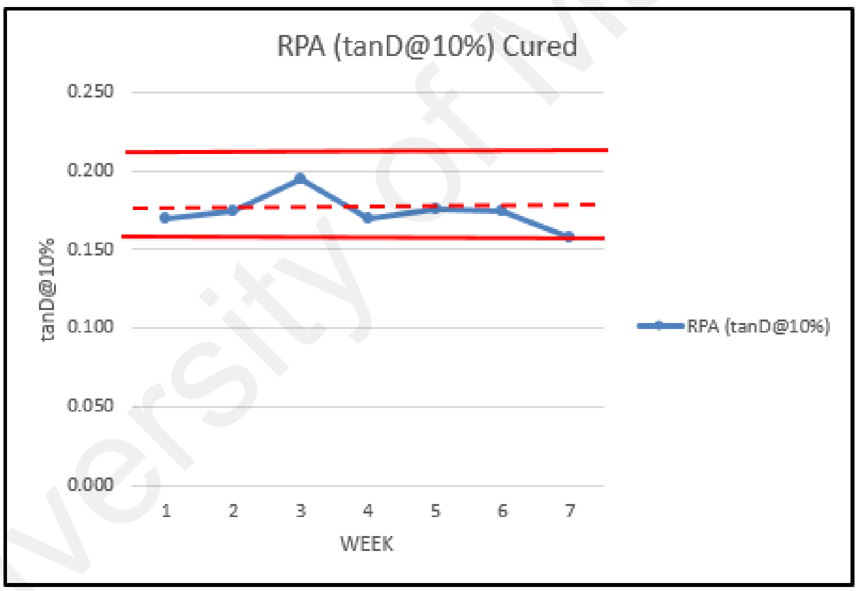


Figure 4.12 RPA (tanD at 10%)

4.2 Correlation results

Table 4.2 showed the summary of results of Spearman Correlation of physical properties to time.

Table 4.2: Spearman Correlation of physical properties to time

	Spearman Correlation	P value
Rheological Properties MDR160		
T10 MDR160	0.3929	0.3833
T40 MDR160	0.3784	0.4026
T90 MDR160	0.5946	0.1591
Mooney Viscosity	0.9643	**0.0005**
Tensile	-0.2143	0.6445
Elongation	-0.2143	0.6445
Modulus	-0.0721	0.8780
Hardness	0.5766	0.1754
Rebound	0.1071	0.8192
RPA		
RPA (G’ at 1%)	0.7857	**0.0362**
RPA (G” at 100%)	0.7500	**0.0522**
RPA (tanD at 10%)	-0.2000	0.6672

CHAPTER 5: DISCUSSION

5.1 Influences of Rheometer Properties MDR160

In this experiment, there was no difference in the rheometer properties throughout seven weeks of ageing time. The amount of accelerator in this recipe was sufficient enough to remain in range the process ability properties especially in T10 (rubber processing time) and T90 (curing time) that controlled by sulphur and zinc oxide. The application of retarder in the recipe will give impact on the processing side, which extend the T10 and scorch time of rubber compound up to 10 – 15% (Frederick Ignatz-Hoover, 2004). Extension of the ageing time and fluctuation in storage temperature and humidity probably will lead to pre crosslinking situation on the compound then affect the curing time.

5.2 Increasing Trend of Mooney Viscosity

The concentration of rubber compound can be determined by measuring the viscosity. This study showed significant positive correlation of mooney viscosity through seven weeks of ageing time. Numerous factors had influenced on the money viscosity trend include; storage area and high humidity. Regarding the extrusion process, high mooney viscosity can cause tearing issue of product after extrusion. High mooney viscosity may lead to high absorption of temperature during the extrusion process (Menting, 2004). In tires industry, the sidewall is the main part that controlled the flexibility of the tires and can affect the cornering situation. High absorption of humidity to the compound probably would give porosity result thus would impact on weight and profile of the sidewall.

Figure 5.1 Looping issue in cracker mill due to high mooney viscosity

Figure 5.2 Porosity area due to high humidity absorb by rubber compound

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5.3 Tensile and Elongation Effects

This study showed negative correlation between tensile and elongation over time. But the result was not statistically significant. Many factors contribute in variation in tensile strength include materials in the recipe. Usage of the emulsion SBR improved the tensile strength of the compound as compared to the usage of solution SBR. Selecting high grade NBR with higher bound acrylonitrile (ACN) content would give higher compound tensile strength even in ageing time (Anderson, 2011) . During the mixing phase, usage of SBR and BR in the recipe lead to variation in the tensile strength in the present of higher concentration of carbon black. The tensile strength increase if more dispersion of carbon black in BR phase and increase percent of dispersion of carbon black during mixing in Banbury (J S Dick, 1999). Different approach applied in order to get good elongation property. Decreasing the surface area of carbon black was usually been applied to improve the elongation of rubber compound over the ageing time.

5.4 Modulus and Hardness Effects

In this study, there were no difference in modulus and hardness over the ageing time. Modulus commonly effected during mixing of compound in Banbury or excessive heat history in compound where they can reduce cure modulus of compound. In other hand, hardness can be improved by increasing the loading of carbon black that would give higher in hardness value. Another approach commonly applied in rubber industry was by reducing the processing aids that give improvement in hardness property. When there were abnormalities in hardness and modulus, we will refer to density value. In this study density measurement throughout seven weeks of ageing time showed constant result. (refer figure 5.3)

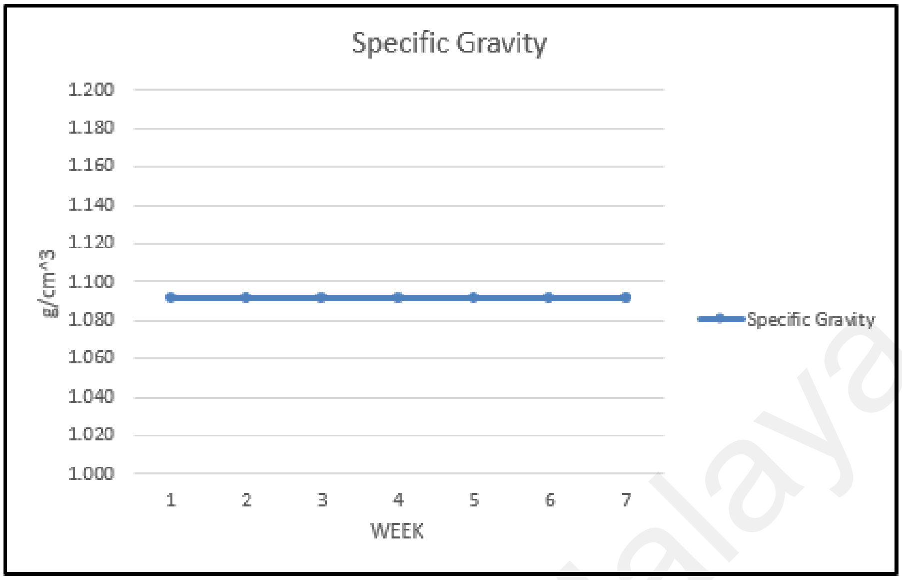

Figure 5.3 Density result

5.5 Rebound Influences

In this study there was no significant difference in rebound properties throughout the ageing time. To get the ultimate rebound property generally, reducing the filler loading such carbon black and silica will give high hysteresis in compound. Rebound can be improved by increasing dispersion during mixing. To get a better dispersion, mixing process need to longer. Another approach to improve rebound in compound is to avoid adding carbon black with oil, stearic acid, or other polar ingredients such as antioxidants because these ingredients may be absorbed into the surface of the carbon black particles, which will interfere with the polymer absorption onto the carbon black surface (Hess W. , 1991). Thus adding oil and other ingredients with the carbon black might interfere with carbon black rubber interaction or the formation of bound rubber. This would increase hysteresis. Therefore, it is better to add the carbon black first before other ingredients to achieve lower hysteresis.

5.6 RPA Effects

Rubber Processing Analyser is to measure the process capability during mixing stage. Most of the data will determine how well the mixing process and interaction of filler-filer during mixing. In this study, there was significant correlation between RPA (G' at 1%) and RPA (G' at 100%) over seven weeks of ageing time.

Common method to get ultimate result in RPA is to prolong the mixing time to get sufficient mixing time and well dispersion between fillers. The result of RPA testing can be used as a baseline data of compound properties at different stages of the rubber process ability. This data is useful in determining the variation of mixing problems, so that the appropriate corrective actions can be taken.

5.7 Limitation

There is no study without limitation. Due to time limitation, we were unable to further extend the time of the experiment to see further physical changes of sidewall compound during ageing. The alternative to this is to use an accelerated oven aging methods but the machine is not available at current setting. Hence the ageing of sidewall compound was tested under ambient temperature.

5.8 Conclusion

Aging of sidewall rubber compound for seven weeks produced significant changes in the physical properties in term of mooney viscosity, RPA (G' at 1%) and RPA (G' at 100%). There were positive correlations between mooney viscosity, RPA (G' at 1%) and RPA (G' at 100%) and ageing time. In contrast, aging sidewall rubber compound for seven weeks did not cause any significant decrease in structural integrity in the rheometer properties, tensile, elongation, modulus, hardness, rebound and RPA (tanD at 10%). Longer time required to see the physical changes during ageing of sidewall rubber compound. Thus, this study should be repeated using accelerated ageing process to see what is the significant changes in the physical properties during sidewall rubber compound ageing process.

CHAPTER 6: REFERENCES

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