Preliminary Study on Effect of Chemical Composition Alteration on Elastic Recovery and Stress Recovery of Nitrile Gloves

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Abstract

Nitrile gloves are widely used in the medical and automobile field due to its superiority in mechanical strength and chemical resistance over natural latex gloves. Natural properties of synthetic latex of nitrile gloves make it stiffer and less comfortable when donned for long periods. Poor elastic recovery of nitrile glove to compressive force also creates an aesthetic issue for customers with high levels of wrinkling after removing from glove box. Aim of this project is to focus on product enhancement to improve elastic recovery and stress relaxation through chemical composition alteration of latex blend. Chemicals such as sulphur, zinc oxide and accelerator that are involved in rubber curing are selected for single factor study to test significance of chemical composition on elastic recovery (%) and stress relaxation (%). Titanium Dioxide is also considered for single factor study due it having filler properties on latex products. Three levels (High, medium, low) of concentration (phr) for all four chemicals is used for single factor study to test significance of chemicals on test results. Elastic recovery measurement using scanner for surface imaging and MATLAB analyzation resulted in data that are non-repeatable and non-significant. Elastic recovery (%) was unable to be proceeded due to limitations to MATLAB analyzation and inconsistent results. Titanium dioxide (TiO_2) showed least significant changes in SR% and mechanical strength data, hence removed from consideration for chemical composition optimization for next phase of study. Stress relaxation (SR%) showed improvement with higher sulphur and accelerator concentration while lower Zinc oxide concentration yielded better SR%. Tensile strength data results were as expected with improvement with higher concentrations of Sulphur, accelerator and Zinc Oxide. But resultant elasticity decreases, shown by lower elongation at break (%).

1. Introduction

Nitrile gloves are synthetic latex gloves also known as Nitrile Butadiene Rubber (NBR) gloves. It is a copolymer derived from 1,3 Butadiene (Petroleum derived) and Acrylonitrile instead of renewable latex source such as Natural Rubber (NR) gloves [1]. NBR gloves are preferred over NR gloves as medical examination due to its superiority in oil resistance, chemical resistance and mechanical strength over NR gloves [2]. Up to 17% of workers in the medical industry suffers from latex allergy which causes itching and drying of skin with similar traits to rashes. This makes nitrile examination glove to mainstay option for medical use.

Several terms are commonly used to describe mechanical properties of rubber, these qualities determine the type and grade of rubber found in the market. Tensile strength is defined as the amount of force (N) per square meter (m) needed to elongate a rubber sample to the point of failure (rupture). Stress Relaxation is defined as the decay of stress under constant strain for a certain period of time due to internal molecular rearrangement (Deformation) to achieve equilibrium with applied force. Rubber with higher tensile strength would require higher load (N) of stress to cause material failure. Elongation (%) is the percentage of stretch of rubber material from its initial state presented in percentage, elongation at break (%) is the measurement of stretch at breaking point of rubber sample. Rubber with high elasticity would display higher elongation (%) when stretched compared to stiffer samples. Elastic modulus also known as Young's Modulus is a measurement of stiffness of a certain material, which is defined as the amount of force exerted at a certain elongation. As such, stiffer or harder rubber samples would display a higher elastic modulus at a certain elongation compared to softer and elastic samples [3]. Rubber Creep is defined as the permanent set of plastic strain experienced by a material under constant strain. Basically rubber samples that have high stress relaxation properties have higher creep when subjected to a certain stress [4]

Issue with Nitrile Glove products

Physical properties between NBR and NR latex have contrasting difference due to their difference in molecular constituents. Natural latex (NR) rubber generally have superior elasticity, resilience and softness compared to NBR, which in nature has higher stiffness, poor elasticity, poor resilience but better tensile strength. The unique characteristics of NBR is linked to the presence of Acrylonitrile (ACN) monomer in its structure, increase in ACN content increases polarity of NBR molecule and glass transition temperature (Tg) properties. Although NBR gloves posses better mechanical properties compared to NR gloves, the stiffer and less elastic nature of NBR gloves do bring about several issues. One issue of NBR gloves is the poor elastic recovery (issue 1) from compressive stress, this results in NBR gloves easily forming surface wrinkles when subjected to compressive force when folded. The other issue regarding NBR glove is its stress retention when worn by any user, the low stress relaxation (Issue 2) property of NBR gloves makes it less comfortable when donned.

This project is a research collaboration between Top Glove Sdn Bhd and Taylor's University to address the two stated issues regarding their Nitrile glove products. The objective is to look into effect of chemical composition optimization to improve stress relaxation and elastic recovery properties. Customer feedback for Top Glove's nitrile glove products has seen customers commenting on the high degree of wrinkling when removed from glove box, which is aesthetically unpleasing as well as causing inconvenience for customers to “open” the gloves up from wrinkled state. The poor stress relaxation of Nitrile gloves makes it less comfortable when donned for long periods as nitrile glove would not conform to the user’s hand. Therefore, a glove that has higher stress relaxation properties would experience deformation (loss of stress) over time and conform to shape of user’s hand giving a better comfort when worn for long durations.

Latex compounding chemicals selected for study

This study aims to look into the effect of chemical composition optimization of four chemicals to enhance nitrile glove elastic recovery from compression and stress relaxation. The four chemicals stated are as stated below:

1. Sulphur (Vulcanizing agent)
2. Zinc Oxide (Vulcanizing activator)
3. Accelerator (Vulcanizing catalyst)
4. Titanium Dioxide (Filler)

Sulphur (S) is the most common vulcanizing agent used in the rubber industry, where it provides sulphur cross-links between long chain polymer molecules which would determine the produced glove properties. Zinc Oxide (ZnO) act as a vulcanizing activator where it would enhance cross-linking between polymer chains and form ionic cross linkage between polymer molecules. Addition of ZnO enhances vulcanizing efficiency and acts as an activator for sulphur crosslinking [5]. Accelerators are also added into NBR latex as vulcanizing catalyst to greatly increase speed of vulcanization with process taking a much shorter time while reducing the use of sulphur in the system [6]. Composition of these three chemicals involved in rubber curing would hold an crosslink density of produced rubber and subsequently the mechanical properties of produced glove [7]. Titanium Dioxide (TiO_2) acts as a rubber filler which reinforces rubber structure for better mechanical properties. Preliminary study will be conducted to test significance of composition change of each chemical towards stress relaxation and elastic recovery of produced latex film. Three of the four chemicals with most significant effect will be studied for optimization to obtain resultant latex with best stress relaxation and elastic recovery properties.

Research Objectives

1. (Preliminary Study) To select three out of the four target chemicals with most significant effect on stress relaxation, tensile strength and elastic recovery properties.
2. To optimize chemical composition of target chemicals to achieve nitrile latex with best stress relaxation and elastic recovery property.
3. To achieve enhanced properties while keeping mechanical properties of nitrile latex within industry standards ASTM D6391 (Table 1.1) for nitrile examination gloves

Table 0.24 ASTM D6319 standard for Nitrile examination gloves

Mechanical Property		**ASTM D6319**
Elongation at break (%)	Before aging After aging	Min 500 Min 400
Tensile Strength (MPa)	Before aging After aging	Min 14 Min 14

Methodology

Chemical composition for significance test

Based on the four target chemicals discussed, composition of chemicals is adjusted to extreme highs and lows to test significance of each chemical towards targeted response (stress relaxation and elastic recovery). Table 2.1 shows the composition of chemicals categorized into three levels. Medium level composition represents normal phr values used in nitrile glove production. “Phr” is an industry standard of denoting chemical ratio based off 100 parts of rubber. when one chemical has its composition varied between high and low values (denoted using **bold** font), all other chemicals phr value is kept at the middle (normal) range to obtain an accurate comparison between produced latex. With this variation, a total of nine batch of latex samples is compounded for single factor study as shown in Table 2.2 below.

Table 0.25 Phr values used for each chemical for significance test

	Low level (phr)	**Medium level – Normal (phr)**	**High level (phr)**
Accelerator	0.1	0.5	3.0
Sulphur	0.2	1.5	3.0
Zinc Oxide	0.1	1.5	5.0
Titanium Dioxide	0.2	1.6	3.0

Table 0.26 Phr values for all target chemicals for each batch of variation in single factor preliminary study.

No.	**Sample name**	**Accelerator (phr)**	**Sulphur (phr)**	**ZnO (phr)**	**TiO_2 (phr)**
1	**Normal**	0.5	1.5	1.5	1.6
2	**High ZnO**	0.5	1.5	**5.0**	1.6
3	**Low ZnO**	0.5	1.5	**0.1**	1.6
4	**High Sulphur**	0.5	**3.0**	1.5	1.6
5	**Low Sulphur**	0.5	**0.2**	1.5	1.6
6	**High accelerator**	**3.0**	1.5	1.5	1.6
7	**Low accelerator**	**0.1**	1.5	1.5	1.6
8	**High TiO_2**	0.5	1.5	1.5	**3.0**
9	**Low TiO_2**	0.5	1.5	1.5	**0.2**

Chemical weight calculation

Calculation of weight of each chemicals (g) requires individual chemical’s parts per hundred (phr) information and Total solid content (TSC%). The information can be subbed into (Equation 1) shown below to calculate chemical weight (g) of each individual chemical. Latex weight is calculated based on 1000g of final solution with final solution of 19% of TSC.

Please note that TSC and Phr information for chemicals in Table 4 is not included for confidentiality reasons

$$\text{Chemical weight (g)} = \frac{\text{chemical phr} \times \text{Latex TSC (\%)} \times \text{Latex weight (g)}}{\text{Chemical TSC}} \qquad (1)$$

Latex blend compounding
Latex compounding involves constant stirring of latex and added chemicals for a period of 24-48 hours to produce a homogenized solution. This step is important in order to avoid settlement of high density solid particles, which may cause uneven properties throughout surface of produced latex. For this study, several conditions are set to be constant for all compounding batches to standardize the compounding process. 24 hour period for latex compounding is chosen based on studies done by Ruslimie et.al [8] showing no increment in crosslink density (mole/g) and tensile strength (MPa) for produced latex with pre-vulcanization time over 24 hours.

1. Compounding period = 24 hours
2. Compounding temperature = Room temperature (25°C)
3. Compounding conditions = Closed conditions
4. Stirrer rotation speed = 600rpm

Manual plate dipping
Lab scaled glove dipping process is done in smaller scale using 1.0 kg compounded latex solution and flat plate former instead of larger sized glove formers which require at least 10kg drum of solution for effective dipping. Former dipping conditions are help similar to nitrile glove production line conditions. Latex samples have to be cooled 24 hours before conducting any test.

1. Former temperature before coagulant dip: 55°C – 60°C.
2. Coagulant temperature: 55°C
3. Coagulant drying conditions: 120°C, 2 minutes
4. Former temperature before latex dip: 60°C – 65°C.
5. Latex drying conditions: 120°C, 15 muntes.

Elastic recovery from compressive stress
An important note is that there is no industrial standardized method of measurement of surface wrinkles through surface imaging or elastic recovery from compressive force for thin rubber films such as glove samples. Method of surface imaging and surface wrinkle analyzation discussed below represents a new and untested method developed through collaborative efforts between Ms. Eunice Phang Siew Wei (Program director/Senior lecturer of Taylor's University) and industrial supervisors at Top Glove Sdn Bhd. This newly developed method can be categorized into 3 stages:

I. Compressive folding of latex sample (Uniform and non-uniform folding)
II. Surface image scanning using a scanner
III. Analyzation and calculation of surface wrinkles (%) through MATLAB

Produced latex samples are folded using two different methods (Figure 2.4.1) to compare which method provided substantial improvement in elastic recovery after a certain period. Surface imaging for samples are scanned for surface morphology analyzation using MATLAB to determine which method is best suited.

Figure 0.29: Standardized folding method (Left) and non-uniform crumpling method (Right)

Several conditions have to set constant for compression of latex films for both methods.

A. (Standard Folding method) each fold is 1.5cm apart measured from the top.
B. (Standard and non-uniform method) Surface imaging on surface area is only applied on area measured 13cm from top. Surface below the 13cm line is not considered.
C. All compressed film is placed under flat surface with a 2.5 kg weight placed on top
D. Compression time is set at 18 hours under the 2.5kg weight.
E. Once weight is removed, surface imaging is done on initial stage (t= 0 minute) and after 10 minutes of recovery (t=10 minute)

MATLAB surface wrinkle analysation

Surface area selection for wrinkled/folded latex samples were manually adjusted using rectangular (blue) box as shown in **Figure 2.4.2** below. Surface area selection was only done within the compounds above the indication line (13cm from top). Cautious manual adjustments were needed to ensure no surface area outside of glove sample is included to avoid error values in wrinkle analyzation results. Image processing using MATLAB results in contrasting of wrinkled surface (white region) and unwrinkled surface (black region) shown in Figure 2.4.2. Wrinkled surface area (white region) over total selected surface is calculated in MATLAB and displayed as wrinkled percentage (%). Calculation of elastic recovery for 10 minute duration is shown in Equation (2)

$$\text{Elastic Recovery (\%)} = (\text{percentage of wrinkles, } t=0) - (\text{percentage of wrinkles, } t=10) \quad (2)$$

Figure 0.30: Selection of measurement area (Blue box) for MATLAB (left)
Contrasting of scanned image (right)

Stress Relaxation and Mechanical property test

Preparation of tensile test piece

All latex test pieces are cut based on ASTM D412 standard as shown in **Error! Reference source not found.** below. Dimensions shown are in units of millimetres (mm). **Error! Reference source not found.** (left) shows the difference in size of die cutter based on different industry standards. Produced latex samples are placed on a flat surface with die cutter placed on top of latex surface. Samples are then placed under an air-pressurized compressor to cut out samples based on die cutter size.

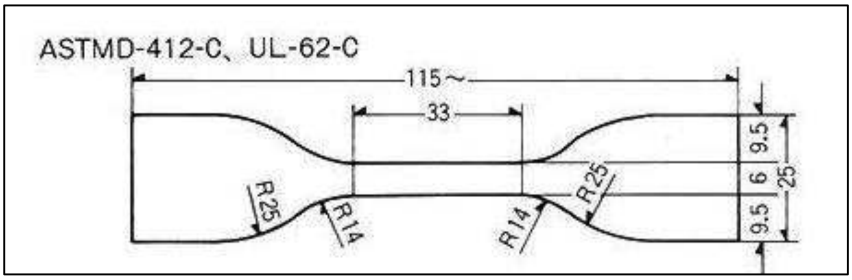

Figure 0.31 ASTM D412 Type C die cutter size [9]

Tensile strength test

Dumbbell shaped test samples were gripped on onto clips of the Universal tensile machine (Figure 2.2). **Standard ASTM tensile test before aging** standard was used, with upward tension force of 500mm/min set for all test samples. Test piece was elongated until sample gage area (middle) snaps at maximum load. Relevant results from test report for this study include:

1. Tensile strength (MPa)
2. Elongation at breaking point (%)
3. Tensile strength at 300% elongation (MPa)

Figure 0.32 Elongation of samples during tensile strength test

2.5.3 Stress Relaxation test

Universal tensile machine is used for conducting stress relaxation. Samples were gripped at both ends to clips on the test machine and elongated upwards for a length of 25.414mm. Samples was left in elongated state for a standard of 6 minutes for all samples. Stress decay of samples throughout 6 minutes with initial stress and final stress values recorded in test report with calculated Stress recovery (%). Equation (3) shows formula of calculation for stress relaxation (%).

$$Stress\ Relaxation\ (\%) = \frac{Fo-Ft}{Fo}\ x\ 100\% \qquad (2)$$

Fo = Initial stress value at t=0

Ft = Stres value after certain duration, t

Results and Discussion

Preliminary study for chemical significance test is a simple test to compare the significance of the four target chemicals (Sulphur, Zinc Oxide, Accelerator, Titanium Dioxide) towards the two target responses of this study (Elastic Recovery and Stress Relaxation%). Collected results are compared, to choose 3 (out of 4 target chemicals) chemicals with most significant effect on both responses when chemical composition is adjusted to very high or very low values. Chosen chemicals are selected to conduct the next stage of full factorial design and chemical composition optimization.

Elastic Recovery

Table 3.1 below shows the elastic recovery (%) of latex films measured at 0 minutes and 10 minutes for both standardized and non-standard folding method.

** elastic recovery measurement was not continued with samples from other batches (High ZnO, Low ZnO, High Accelerator…) due to results shown being unrepeatable**

Table 0.27 Elastic recovery percent of latex film for differnt folding method.

Sample Name	**Wrinkle percentage %**					
	Standard folding			**non-standard folding**		
	t=0	**t=10**	**Recovery%**	**t=0**	**t=10**	**Recovery%**
High Sulphur 1	34.2721	26.3442	7.9279	34.1373	40.3772	-6.2399
High Sulphur 2	53.976	54.1037	-0.1277	54.272	53.5781	0.6939
High Sulphur 3	30.5375	31.5073	-0.9698	44.2976	44.7708	-0.4732
High Sulphur 4	16.7468	22.1309	-5.3841	54.6769	50.4428	4.2341
Low Sulphur 1	51.0367	57.8281	-6.7914	45.9153	45.1422	0.7731
Low Sulphur 2	51.3293	52.9274	-1.5981	46.7022	46.8926	-0.1904
Low Sulphur 3	38.0716	32.7805	5.2911	47.1178	46.1577	0.9601
Low Sulphur 4	30.0462	25.8878	4.1584	39.0742	39.1272	-0.053

Test results as shown in Table 3.1 were clearly non-repeatable and non-sensible. Although MATLAB was not able to differentiate elastic recovery in 10 minutes, the minor differences can be seen through visual judgement based on scanned images shown in **Error! Reference source not found.** below. There is a certain amount of recovery in wrinkling depth between both samples. The scanned image after 10-minute of recovery (right) is bigger in terms of surface area compared to sample image at 0-minute due to the minor elastic recovery in wrinkle depth causing latex sample to slightly "expand" in size as it recovers. Wrinkled samples would display higher degree of elastic recovery after longer amounts of time, but that would beat the purpose of the objective, as end users were dissatisfied with the high number of wrinkles when gloves were immediately removed from glove box (Elastic recovery in short period)

Figure 0.33 Scanned images of latex surface at 0 minutes (left) and 10 minutes (right)

Hence the issue with results being non-sensible (negative values) and non-reproducible can be summarized in 2 major factors:

1. MATLAB analyzation limitations

As discussed with the industrial supervisor who wrote the coding, MATLAB was only able to contrast wrinkled (white) and unwrinkled regions (black) as shown in Figure 2.2 and calculate total surface area of wrinkled regions (white) over total selected surface area. But based on visual differences shown in Figure 3.2, regions of wrinkle lines and regions remains largely the same for samples at 0-minute and 10-minute. Only a change in depth of wrinkles were observed. Hence MATLAB was not able to distinguish the differences between both images making results insignificant. Future improvements of coding may allow or wrinkle depth to be differentiated using MATLAB.

2. Inconsistency in manual adjustment for selected surface area

As shown in Figure 2.2, selection of surface area is done by manually adjustments of the blue indication box. This adds in factor or inconsistency when surface area of 0-minute sample and 10-minute sample are selected. Since wrinkle regions show no difference in MATLAB between both images, difference in selected area may cause an increment or decrease in resultant elastic recovery (%). This explains the negative values of elastic recovery (%) shown in **Error! Reference source not found.**. Future improvements can be done in coding to automatically identify and select area of latex sample to avoid such consistency.

Significance test for stress relaxation and mechanical strenght

Target chemicals (Sulphur, Zinc Oxide, Accelerator, Titanium dioxide) have phr adjusted beyond normal production range (Refer to Table 2.1) to select three chemicals with most significant change in stress relaxation (%) response. Table 3.2 below shows stress relaxation (%) results of each chemical variation. All data shown for stress relaxation and tensile strength were averaged out between data obtained from 4 test samples of the same composition batch. Table 3.3 below shows the tensile strength, elongation at break (%) and elastic modulus at 300% elongation data for the same samples.

Table 0.28 Stress relaxation (%) after 6 minutes of constant strain

	Stress at 0 minute, *Fo* (MPa)	**Stress after 6 minutes, *Ft* (MPa)**	**Stress Relaxation (%)**	**Difference between High and Low formulation (%)**
High Sulphur	1.861	0.856	42.58	3.40
Low Sulphur	1.696	0.722	45.98	
High TiO_2	1.664	0.734	44.10	0.47
Low TiO_2	1.405	0.626	44.57	
High Accelerator	1.708	0.789	41.38	4.81
Low Accelerator	1.553	0.643	46.19	
High Zinc Oxide	0.921	0.338	36.76	13.03
Low Zinc Oxide	0.500	0.250	49.79	

Table 0.29 Tensile strength, Elongation at break(%) and Elastic modulus result for test samples

	Tensile Strength (MPa)	Difference between High and Low formulation (MPa	Elongation at break (%)	Difference between High and Low formulation (%)	Modulus at 300% Elongation (MPa)	Difference between High and Low formulation (MPa)
High Sulphur	46.70	23.71	460.8	73.9	12.05	6.49
Low Sulphur	22.99		534.7		5.56	
High TiO_2	21.44	1.45	481.37	26.93	6.64	1.18
Low TiO_2	22.89		508.3		5.46	
High Accelerator	23.62	2.95	473.8	94.37	7.37	2.42
Low Accelerator	20.67		568.17		4.95	
High Zinc Oxide	24.60	8.18	304.37	213.68	22.17	20.08
Low Zinc Oxide	14.19		518.05		2.09	

Titanium dioxide (TiO_2) which acts as a reinforcement filler, did not have significant effect in stress relaxation (%) when concentration is adjusted to extreme highs and lows. The other three target chemicals (Sulphur, Zinc Oxide, Accelerator) were directly involved in rubber curing process, change in concentration in these chemicals would have a significant impact in crosslink density of produced latex. Stress relaxation (%) between High concentration (3.0 phr) and low concentration (0.2 phr) only resulted in a difference of 0.47 MPa. The insignificant difference for high and low samples of TiO_2 in stress relaxation (%) can be backed up by mechanical test data shown in Table 3.3, which shows the least difference across the board for tensile strength, elongation at break (%) and elastic modulus at 300% elongation. Data shown in this table further proves that a direct influence in crosslink density can effect mechanical strength and overall stiffness of a latex sample.

An additional observation can be made in Table 3.2, stress relaxation (%) high and low concentration of Zinc Oxide (ZnO) shows an exceptionally higher difference compared to other chemicals, with difference of 13.30% in stress relaxation. This clearly shows that ZnO is the most significant factor involved in enhancing stress relaxation in produced latex. This may hint that ionic crosslinking in rubber molecules can significantly impact mechanical properties of produced rubber. By referring to results in Table 3.3, elongation at break (%) High and low Zinc Oxide samples show the highest difference of 213.68% between samples from both batch. The same trend is seen with elastic modulus at 300% elongation. High ZnO samples had exceptionally high elastic modulus at 22.17 MPa while low ZnO samples were exceptionally low at 2.09 MPa. This meant that low ZnO samples were exceptionally soft, with much lesser load (N) needed to stretch samples to 300% elongation. Based on discussions with industrial supervisors on the relation between all these data, a summarization between the relationship of mechanical properties can be summarized below in Table 3.4 below:

Table 0.30 Relationship between mechanical properties in gloves

	Tensile strength (MPa)	**Elongation at break (%)**	**Elastic Modulus – Rubber Stiffness (MPa)**
Higher Stress Relaxation (%)	Lower	Higher	Lower Stiffness
Lower Stress Relaxation (%)	Higher	Lower	Higher Stiffness

Results from preliminary study of significance in effect of chemicals can conclude two major statements:

1. Titanium dioxide (TiO_2) was the least significant chemical towards stress relaxation (%). It will be excluded for full factorial design and composition optimization.

2. Zinc Oxide (ZnO) is the most significant factor affecting stress relaxation (%) and overall softness of produced latex.

4. Conclusion

As it stands, elastic recovery measurement through surface imaging using scanner shows that the method is not suited for this project as results obtained are inconsistent and non-repeatable. MATLAB analyzation of surface wrinkle was only able to determine the number of wrinkles / wrinkled regions of selected area but unable to distinguish the depth of surface wrinkles on nitrile latex surface. Elastic recovery of wrinkled films had recovery of wrinkled depth but little to no recovery from number of wrinkles after 10 minutes of recovery period. Hence, MATLAB was unable to distinguish difference between sample image at 0 minute and 10 minute. Manual selection of analysed surface area through manual adjustment also caused inconsistency in selected areas for 0 minute and 10 minutes samples, resulting in some samples yielding negative elastic recovery values. Titanium Dioxide (TiO_2) showed least significant changes for SR% and mechanical properties between high-low concentrations and between normal formulation samples. Increment in sulphur and accelerator concentration caused an increment in SR%, possibly due to increased sulphur crosslinking in samples. But increment of Zinc Oxide caused decrease in SR% due to ionic crosslinking between rubber molecules. Tensile strength results show an expected outcome with an increment in mechanical strength with higher concentrations of sulphur, accelerator and zinc oxide due to higher density crosslinking. But enhanced mechanical strength had a resultant effect of decreased elasticity of produced samples, with elongation at break (%) showing decreased value with higher concentrations. Sulphur, accelerator and Zinc Oxide are chosen for full factorial study for chemical composition optimization for next phase of the study.

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