

5-2014

# Introduction of Natural Oils into Rubber Compounds

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# Introduction of Natural Oils into Rubber Compounds

Thesis submitted in partial fulfillment of Honors

By

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April 1, 2014

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## Table of Contents

Abstract.....	1
Introduction.....	1
Literature Comparison:.....	3
Materials and Methods.....	7
Results.....	19
Discussion.....	33
Conclusion.....	41
References.....	44

## List of Tables

Table I Chemical Profile of the Ford Motor Co. candidate Natural Oils.....	4
Table II Sample Rubber Formulation with Natural Oil (Ford Motor Co.).....	5
Table III Natural Rubber Materials and Reagents (HEXPOL).....	9
Table IV PolyChloroprene Materials and Reagents (HEXPOL).....	11
Table V Ethylene-Propylene Diene Monomer (EPDM) Grade E Materials and Reagents (HEXPOL).....	11
Table VI Styrene Butadiene (SBR) Materials and Reagents (HEXPOL).....	12
Table VII Nitrile Materials and Reagents (HEXPOL).....	12
Table VIII Natural Rubber (Mooney Viscometer) Results.....	19
Table IX PolyChloroprene (Mooney Viscometer) Results.....	19
Table X EPDM (Mooney Viscometer) Results.....	20
Table XI Styrene Butadiene (Mooney Viscometer) Results.....	20
Table XII Natural Rubber (ODR) Results.....	21
Table XIII PolyChloroprene Rubber (ODR) Results.....	21
Table XIV EPDM Rubber (ODR) Results.....	21
Table XV Styrene Butadiene Rubber (ODR) Results.....	22
Table XVI Natural Rubber Tensile Results.....	22
Table XVII Polychloroprene Rubber Tensile Results.....	22
Table XVIII EPDM Rubber Tensile Results.....	23
Table XIX Styrene Butadiene Rubber Tensile Results.....	23
Table XX Natural Rubber (Durometer & Specific Gravity) Results.....	24
Table XXI Polychloroprene Rubber (Durometer & Specific Gravity) Results.....	24
Table XXII EPDM Rubber (Durometer & Specific Gravity) Results.....	24
Table XXIII Styrene Butadiene Rubber (Durometer & Specific Gravity) Results.....	25
Table XXIV Natural Rubber (Compression Set) Results.....	25
Table XXV Polychloroprene (Compression Set) Results.....	26
Table XXVI EPDM Grade E (Compression Set) Results.....	27
Table XXVII Styrene Butadiene Rubber (Compression Set) Results.....	28
Table XXVIII Natural Rubber (Aged Tensile) Results.....	29
Table XXIX Polychloroprene Rubber (Aged Tensile) Results.....	30
Table XXX EPDM Grade E (Aged Tensile) Results.....	31
Table XXXI Styrene Butadiene (Aged Tensile) Results.....	32
Table XXXII Natural Rubber Candidate Oil Results.....	42

Table XXXIII Polychloroprene Candidate Oil Results .....	42
Table XXXIV EPDM Candidate Oil Results .....	43
Table XXXV Styrene Butadiene Candidate Oil Results .....	43

**List of Figures**

Figure 1 Lab Mixer Diagram .....	9
Figure 2 Mooney Viscometer and Rotor Diagram .....	14
Figure 3 ODR Diagram, Rotor Cavity, and Graph of Sample.....	15
Figure 4 Tensometer and Dumbell Example .....	16
Figure 5 Type A Shore Durometer .....	17

## **Abstract**

In the rubber industry, plasticizers for rubber compounds mainly consist of petroleum derivatives. Consequently, the rubber industry is in constant competition with many petroleum consumers. This competition places an economic strain on rubber companies such as HEXPOL RUBBER COMPOUNDING L.L.C. In order to alleviate this strain, natural oil alternatives to petroleum plasticizers are of novel research interest and are investigated in this thesis project.

## **Introduction**

Plasticizers are used in rubber chemistry to soften the rubber compounds to ensure thorough mixing of the compound and easy processing of the finished rubber compound in a factory setting. Depending on the rubber compound's application, the type of oil used as a plasticizer may affect the physical properties such as the hardness of the compound. Most of the current plasticizers used today consist of naphthenic and paraffinic petroleum-based oils. A naphthenic oil is defined as any oil predominately composed of cycloaliphatic rings of various types with some aromatic and aliphatic substituent. The core of the molecule is represented by the cycloaliphatic moiety.<sup>1</sup> A paraffinic oil is defined as any oil composed primarily of various alkanes.<sup>2</sup>

The goal of a plasticizer is to provide ease of flow because polymers that make up the primary linking force in a rubber compound are resistant to flow.<sup>3</sup> The term flow describes how the polymer responds after it is exposed to heat and a pushing force. The polymer itself may flow well at very high temperature, but this will initiate cross-linking in the rubber matrix. The result of cross-linking at high temperature produces bonds between the individual polymer strands. This creates the finished product that companies sell as their final parts. In order for this compound to process well it must have addition of an oil. The chemicals being used in the rubber

compound must be taken into account when trying to improve the flow of the rubber compound are the chemicals being used in the rubber compound. If the wrong oil is used, the oil may appear on the rubber's surface. The result of this would be an unwanted compound, which has little use in this research project.

In a formulation of a rubber compound, the overall chemical structure of the desired polymer is used to determine what oil the rubber chemist will choose as a plasticizer. There are other options besides paraffinic and naphthenic oils such as: aromatics, castor oil, and ester plasticizers.<sup>4</sup> The petroleum oils listed previously are plasticizers for polymers such as: butyl, styrene butadiene, and nitrile polymers. Castor oil is a common plasticizer of butyl rubber. Castor oil is renewable and very little research has been done on this polymer. On the other hand, styrene-butadiene and nitrile polymers both use petroleum based plasticizers. Styrene -butadiene has a high degree of unsaturation, so it works great with aromatic oils. Nitrile polymers will not work well with any traditional oils due to the polarity of the pendant nitrogen group in the polymer. Instead, ester plasticizers are introduced to this compound to improve processability.<sup>5</sup>

The following are trade names of petroleum based oils used in this study: Sunpar 2280 Liquid, SI-69 Liquid, Polycizer Butyl Oleate, Sundex 790 T Liquid, Calsol 8240, and Plasthall P-643. These oils are mainly produced as by-products from the petroleum refining industry, and this creates an issue for the rubber industry. Competition is high between fuel companies who need this petroleum for their refining processes, and the rubber companies such as Goodyear, Cooper Tire, and Firestone who use the by-products as plasticizers. Many rubber companies are now looking into alternatives that are both renewable and effective in rubber compounds being produced.<sup>6</sup> There are many renewable oils available in the world today, but they must be low

cost, sustainable, and meet rubber compound requirements to be viable plasticizers in the rubber industry. These are issues that rubber chemists and researchers are trying to address in research.

### **Literature Comparison:**

Until a few years ago, not many companies in the rubber industry found it necessary to investigate the introduction of renewable plasticizers into their large scale operations. Due to the climb in petroleum costs and rush of the green chemistry movement, rubber companies feel extreme pressure to begin research in this area. There are many branches of rubber chemistry around the world including: custom, tire, hose, and aerospace mixing. Each company has their own way of doing things, so it is the responsibility of each research and development facility to conduct research in this area. Some companies, or independent research facilities, have released details on their research on natural oil alternatives to better outline a project for future researchers.

A main thing that researchers look at during a study like this, is how the natural oil interacts with the rubber matrix. Plant oils can be characterized by their fatty acid distributions, which determines the relative level of unsaturation in the oil.<sup>7</sup> A correlation can be drawn between the relative level of unsaturation and the compatibility of the rubber. If one uses a highly unsaturated oil with an ethylene propylene diene monomer polymer (EPDM), it would result in mixing and processing issues. This is because the chemical nature of EPDM does not contain many double bonds. The common rule in rubber chemistry is to match the oil with the chemical structure of the polymer. For example, in EPDM it would be best to use an oil with little to no double bonds because this would be most compatible with the polymer. So, the selection of oils must be diligent and selected with evidence proving exactly why this oil fits the specific polymer.

The Ford Motor Co. research group did a study on the introduction of several different natural oils into styrene-butadiene rubber (SBR) tire tread compounds and natural rubber (NR) sidewall compounds. The oils chosen in this study were palm, high linolenic flaxseed, and low saturated soybean oils. Fatty acid profiles of these oils were taken and are listed in Table I.<sup>8</sup> Table I provides a display of the nature of the natural oils before they were implemented into Ford Motor Co.'s rubber compounds. Some fatty acids interact well with the rubber and others may not. Depending on the interactions, this tells the rubber chemist just how viable these oils are through experimentation.

Fatty acids distributions are displayed in Table I as percentages. Table I provides a comparison between the candidate oils.<sup>9</sup> The percentages vary upon the crop source and processing methods. For example, low saturated soybean oil was selected based on its promising results in previous studies with degummed soybean oil.<sup>10</sup> The level of saturation in low saturated soybean oil about 7 percent compared to 15 percent in traditional soybean oil. The other oils were also selected based on their chemical make-up. After selection, the oils must be formulated into recipes, mixed, and testing must be done.

**Table I Chemical Profile of the Ford Motor Co. candidate Natural Oils**

Chemical Structure (Carbon-Carbon Double Bonds)	Fatty Acid	Palm Oil	High Linolenic Flaxseed Oil	Low Saturated Soybean Oil
C 16:0	Pamitic	44	4.89	4.07
C 18:0	Stearic	4.5	2.23	3.21
C 18:1	Oleic	39	9.54	21.71
C 18:2	Linoleic	10	12.76	60.36
C 18:3	Linolenic	1	69.08	8.7

In Table II, a general recipe is given for better clarification. Table II is the basic layout for everything that goes into a typical tire tread compound. The only thing that was changed throughout this study was the processing oil. The mixing protocol that they chose for this study is called a “masterbatch mixing cycle”.<sup>11</sup> The reason that this was chosen was to ensure that all



the ingredients in the recipe are thoroughly mixed. Also this ensures good testing results. The compound was mixed by Ford Motor Co. three times in the following set of steps. Ford Motor Co. combined the elastomers, silica, TESPT, and other chemicals. After the initial chemical materials were added the stearic acid, zinc oxide, and the processing aid were incorporated into the mix. Finally, the combined accelerators and sulfur were added to complete the mixing cycle.<sup>12</sup> All of the batches were mixed, then tests were performed on the various iterations of this tire tread compound. This is done in almost all studies pertaining to novel natural oil plasticizers.<sup>13</sup>

**Table II Sample Rubber Formulation with Natural Oil (Ford Motor Co.)**

**\*Rubber formulation, parts per hundred rubber (phr), by weight.**

Formulation	
Component	phr
S-SBR, OE	84.78
S-SBR, Clear	18.34
Natural Rubber	20.00
N234 Carbon Black	10.00
Zeosil 1165 MP	60.00
TESPT coupling agent	4.80
Processing oil	10.00
Microcrystalline Wax	2.00
Antiozonant	2.00
Antioxidant	0.50
Zinc Oxide	1.90
Stearic Acid	1.50
Sulfur	1.50
Sulfenamide Accelerator	1.30
Guanidine Accelerator	1.50
Total phr	222.12
OE = Oil Extended TESPT = bis(triethoxysilylpropyl) tetrasulfide N234 = Relates to the carbon black pellet size MP = Micro-Pearl	

Mooney viscosity measures the amount of torque generated by a (27-30g) sample when a rotor is rotating at a speed of 2 rpm.<sup>15</sup> The viscosity of the compound helps one decide what size rotor to use, but traditionally a large rotor is used. In a study of natural oils as plasticizers

conducted by University of Sri Jayewardenepura used a standard sample size given previously and a large rotor was used with the natural rubber sample.<sup>16</sup> Another study done by Kuriakose A.P. & Varghese M. used a large rotor due to the low viscosity of polychloroprene rubber.<sup>17</sup> Many rubber compounds will allow the use of a large rotor in the Mooney Viscometer. It is only the sample that exceed the machine's maximum torque limit of 200 Mooney Units, then a small rotor is used.<sup>18</sup>

Mooney scorch is conducted in the same instrument as Mooney viscosity testing, which is the Mooney viscometer. Mooney scorch has a different goal because it is trying to measure over a period of constant temperature, pressure, and rpm the cure rate of a compound. When a rubber compound is exposed to high temperature for a set period of time, the crosslinking agents begin to form crosslinks in that polymer.<sup>19</sup> The compound's characteristics and potency of the cross-linker, dictate how fast or slow the rubber compound reaches maximum torque. In the machine there will be a curve given and at the time the sample reaches its minimum the machine takes a reading, and for each unit (T1, T3, and T5) the instrument takes a reading. The instrument reads the time it takes for the rubber compound to increase one, three, and five units from the initial minimum reading (ML). This tells a researcher approximately how much time in the factory setting they have to process the rubber compound.

The Oscillating Die Rotor (ODR) testing takes an accurate reading of the rubber compound curing characteristics. This is displayed by a curve and different readings are taken by the machine to characterize the individual samples. This machine measures the ML, MH,  $t_{s2}$ , and  $t_{c90}$ . These are the most important readings taken by the ODR curemeter. The ML is the samples minimum reading and MH is the highest reading. The  $t_{s2}$  is the time it takes the compound to increase 2 units from its ML reading. The  $t_{c90}$  is the time the compound takes to reach 90% of its

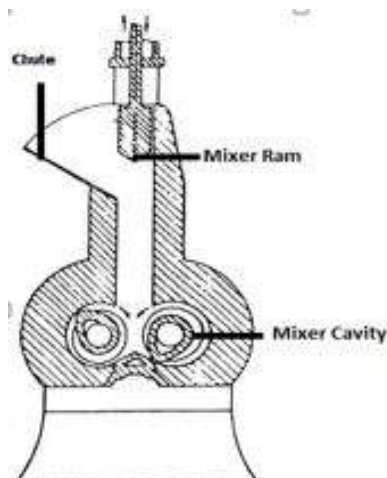
maximum torque reading. With this in mind,  $t_{c90}$  assists in determining production cure temperatures of the novel compounds. The ideal  $t_{c90}$  measurement is one that allows the producer the maximum production output with little error in a factory setting.

Physical testing and heat aging are two very popular ways of testing the sample's final viability. Physical tests include the durometer that measures the hardness of the compound. The tension test measures several characteristics of the compound after it has been cured in a lab press under constant temperature and pressure. The typical testing for tension is given by the ASTM D412 testing method, which defines the parameters of the test. Heat aging and compression set are two tests that measure the sample's resistance to degradation by a hot air oven. Testing parameters are given by the ASTM D412 and ASTM D395. These testing methods are used by all researchers in the rubber industry due to their ease of repeatability.

For example, in a study done with rice bran oil in tire tread compounds the same parameters explained above on this page were followed for testing, and the only thing that differed was the mixing procedure. In this study, all reagents except curatives, were added in the first step then, sulfur and accelerators were added in the second step.<sup>14</sup> The degree of testing that one chooses to do in the lab depends on how thorough one wishes to be with their results. In nine studies conducted on tire tread and sidewall compounds the following instrumentation was used: Mooney viscosity/scorch, oscillating die rotor (ODR), tensile, heat aging, and compression sets. The results were fairly consistent between all of the studies and would be expected to be because producers of the polymers have set parameters for their products. These parameters were discussed in the Results and Discussion section of this thesis.

## **Materials and Methods**

Five compounds of novel interest to HEXPOL RUBBER COMPOUNDING LLC were chosen based on their compatibility with natural oil alternatives. The compounds were already produced in a factory setting, so the weights of their formulations had to be reduced in order to fit into a laboratory mixer. The lab mixer was a miniature version of the factory mixer used in this project. Figure 1, below, contains a diagram of a typical lab mixer. Figure 1 contains a few key features of the lab mixer that was used for the mixing of all compounds during this research. The chute is where all the materials and reagents for each compound were added and it continued down to the mixing cavity. The mixer ram was used to push the ingredients down into the mixing cavity and to keep it there. In order for the mixer ram to do its job, it was pressurized to push and hold all of the materials and reagents in the mixing cavity. This was done by pressurized air that was delivered to the top of the ram. This ensured thorough mixing of compounds unless the weight exceeded what was proper for the lab mixer. The mixer cavity contained two screws that rotated at various RPM, also they rotated in an opposite direction to each other. This enabled tough polymers to be shredded into smaller monomers. Since these polymers were shredded due to mechanical friction, heat was produced in the mixer cavity. Typically, a temperature sensor is placed in the front and back of the cavity to monitor temperature change effectively. Consequently, each compound that was mixed during this research has a different temperature at which it should be dropped out of the bottom of the mixer. The procedure for each rubber compound used in this study will be in Tables III-VII. The previous statement is termed as the compounds “mixing procedure” in which the RPM of the rotors is low at the beginning and slowly increased to reach the compound’s drop temperature.



**Figure 1 Lab Mixer Diagram**

Tables III – VII contain all of the materials and reagents used in this study. The ingredients varied from compound to compound. For example, Table III contains a rubber formulation that has all of the materials and reagents that were used in this particular compound. The polymers in this table include natural, polyisoprene, and polychloroprene. The inert filler may be clay or talc, which is common in the rubber industry. Carbon black simply refers to a reinforcing material added to the rubber, in contrast, processing aids include waxes and other low molecular weight polymers. Stearic acid is an activator in many rubber based polymerization reactions. Petroleum oil is the plasticizer of the rubber compound in this protocol. The natural oils were substituted for the petroleum oils in this study. The petroleum oil used as the control and natural oil alternatives used the same protocols for mixing in tables III-VII.

**Table III Natural Rubber Materials and Reagents (HEXPOL)**

<b>Ingredients (Masterbatch)</b>	<b>Weight (grams)</b>
Natural Rubber	572
Polyisoprene Rubber	123
PolyChloroprene Rubber	123
Inert Filler	245
Inert Filler	81.7
Carbon Black	163
Processing Aid	0.82
Processing Aid	16.3

Processing Aid	4.1
Anti-Oxidant	16.3
Stearic Acid	16.3
Anti-Oxidant	16.3
Petroleum Oil	123
<b>Cross-linking Agents (Cure Pass)</b>	
Accelerator Package	3.0
Sulfur	2.2
Zinc Oxide	6.5
Total Weight	~1500

This specific natural rubber compound contained a step-wise mixing process. The first step is termed the “masterbatch” because it contained all of the reagents excluding the various crosslinking agents or curatives. The curatives are added in the second step of the process commonly termed the “cure pass”. In the masterbatch step, the beginning RPM was 50-60 and the powder reagents and oil were added to the mixer. After about fifteen seconds, the polymers were added to the mixer and a temperature increase was observed due to mechanical friction that produced heat. The ram was pressed down to force any remaining materials or reagents into the mixing cavity. The ram pressure was released at a certain temperature or time intervals termed as a “sweep”. A sweep allowed materials and reagents that had gotten on the top of the ram, to re-enter the mixing cavity, and allowed the compound to “turn over”. The term “turn over” referred to the rotors sometimes keeping unmixed material at the top of the rotors, so this step was employed to ensure thorough mixing.

This masterbatch step was repeated in the order listed: control, palm, soybean, fryer, canola, and safflower oils. The mixer was cleaned to ensure no cross contamination between each of the iterations. The cure pass of this compound was lower due to the cross-linking agents that were in the presence of the polymer. Cross-linking in rubber is temperature sensitive, also an already cross-linked compound would not be advantageous for customer processes. In order to avoid overcuring of the rubber the drop temperature of the cure pass was lower than the

masterbatch. The masterbatch drop temperature was higher, in contrast, with the cure pass that was at a lower temperature. Both of these steps lasted about 2-3 minutes depending on the time it took to reach the drop temperatures, respectively.

**Table IV PolyChloroprene Materials and Reagents (HEXPOL)**

<b>Ingredients</b>	<b>Weight (grams)</b>
PolyChloroprene Rubber	310
PolyChloroprene Rubber	465
Carbon Black	194
Inert Filler	155
Processing Aid	15.5
Inert Filler	31.0
Anti-Ozonant	23.2
Stearic Acid	3.872
Zinc Oxide	46.5
Accelerator Package	15.4
Sulfur	3.8
Crosslinker	23.2
Anti-Oxidant	11.6
Petroleum Oil	213
Total Weight	~1500

The Polychloroprene compound was mixed in a similar manner as the natural rubber compound. The only things that differed in the mixing procedure was a lower drop temperature due to the nature of this polymer. The curatives were added at the beginning of mixing, and cross-linking had begun sooner than in a step-wise process. The mixing in this compound took about 2-3 minutes, which was similar to the latter compound.

**Table V Ethylene-Propylene Diene Monomer (EPDM) Grade E Materials and Reagents (HEXPOL)**

<b>Ingredients</b>	<b>Weight (grams)</b>
EPDM Rubber	195
EPDM Rubber	456
Carbon Black	476
Inert Filler	43.2
Inert Filler	32.6
Cross-Linker	13.7
Processing Aid	6.5
Zinc Stearate	43.2

Zinc Oxide	32.6
Cross-Linker	28.8
Anti-Oxidant	13.0
Petroleum Oil	195.45
Total Weight	~1500

The EPDM rubber followed a comparable mixing procedure to the polychloroprene compound. The drop temperature of this compound was slightly lower, and the compound was mixed thoroughly.

**Table VI Styrene Butadiene (SBR) Materials and Reagents (HEXPOL)**

<b>Ingredients</b>	<b>Weight (grams)</b>
SBR Rubber	650.60
Carbon Black	487.95
Stearic Acid	6.506
Zinc Oxide	26.024
Processing Aid	22.771
Processing Aid	6.506
Anti-Oxidant	22.771
Anti-Oxidant	9.760
Accelerator Package	18.2
Petroleum Oil	244
Total Weight	~1500

The SBR compound mixing procedure was unique from the other rubber compounds. In the masterbatch step the polymer, carbon black, and oil were added. Then, all other powder ingredients were added in the cure pass. This ensured that all of these elements were mixed uniformly, then the cure pass initiated the cross-linking process in the rubber. The drop temperatures for each of the steps were similar to natural rubber compounds.

**Table VII Nitrile Materials and Reagents (HEXPOL)**

<b>Ingredients</b>	<b>Weight (grams)</b>
Nitrile Rubber	577
Nitrile Rubber	144
Carbon Black	505
Stearic Acid	3.6
Zinc Oxide	36.1
Inert Filler	9.4
Anti-Oxidant	21.6



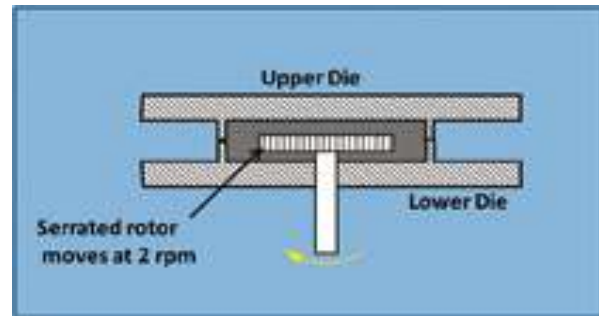
Accelerator/Retarder Package	40.4
Sulfur	2.2
Nitrile Rubber	50.5
Petroleum Oil	108
Total Weight	~1500

The nitrile mixing procedure was similar to the polychloroprene and EPDM rubbers. The control oil for these compounds were mixed. But, all of the natural oil alternatives did not mix. The nature of this incident will be explained in the results and discussion section.

The next set of information contains all of the physical testing that was done on each of the rubber compounds. The physical testing included: Mooney viscosity, Mooney scorch, oscillating die rotor (ODR), tensile, specific gravity, and durometer. Each compound was tested following the pre-set customer specifications for each compound. Consequently, information in the tables varied and contained Mooney viscosity or Mooney scorch data.

A Mooney viscometer was designed for measuring the “shearing viscosity” of rubber materials. The shearing action was performed by a rotating disk in a shallow cylindrical cavity filled with a rubber sample. The rubber sample was cut into two square pieces of a cumulative weight of approximately 25 grams to properly fill the cavity. One piece was placed on the top of the die and the second was placed on the bottom of the die. The rotor containing the sample was placed in the instrument and the testing shield was closed. Figure 2, contains a visual of a typical Mooney viscometer and rotor design below:<sup>20</sup>

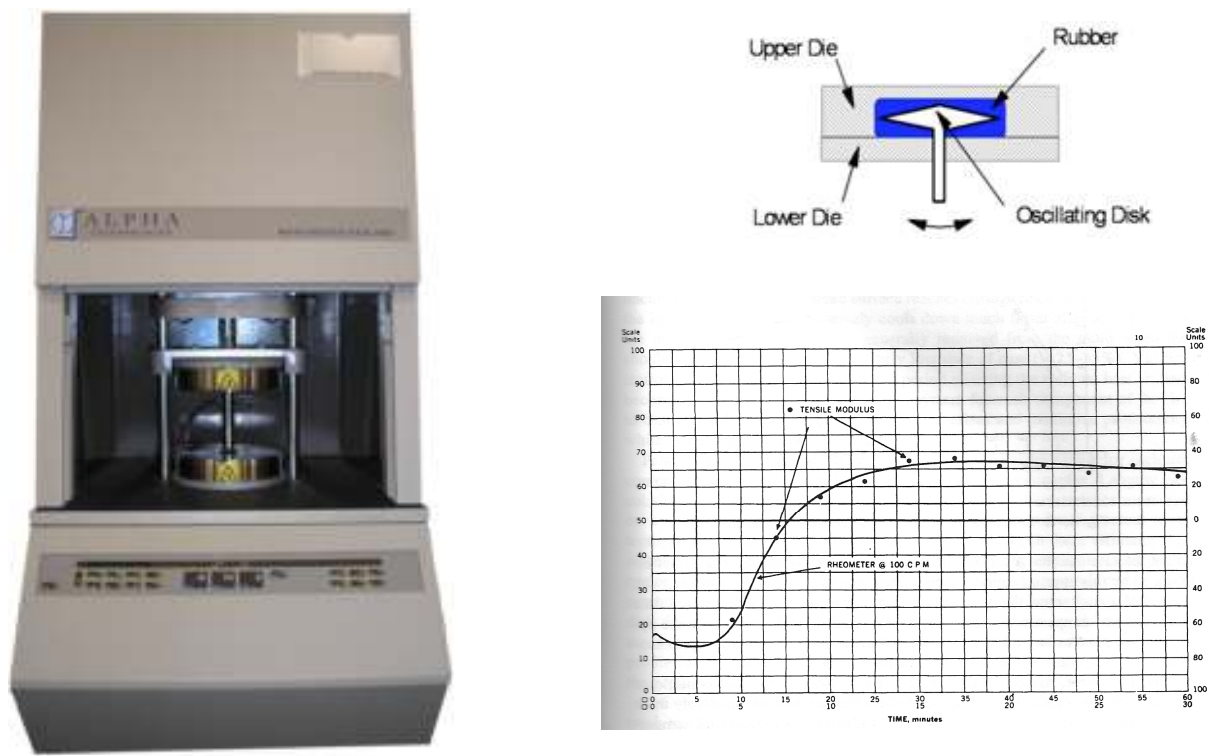
Figure 2 shows a general Mooney viscometer that contained two heated plates that were used to produce the necessary temperature conditions for each of the compounds. The bottom plate contained the rotor and motor that spins the rotor. As seen in the diagram of the rotor the cavity was easily visible to allow all of the rubber to be pressed under constant pressure.



**Figure 2 Mooney Viscometer and Rotor Diagram**

The oscillating die rotor (ODR) instrument produced data differently from the Mooney Viscometer, but still dealt with a rubber sample being pressed into a cavity under constant temperature and pressure. Unlike the Mooney viscometer, the rotor for the ODR was oscillated through a small degree of arc rather than continuously rotated. A rubber sample of about 10-15 grams was placed on the rotor and the sample testing began. The rotor oscillated and the torque required to oscillate the rotor was measured. The process of vulcanization in rubber occurs within the instrument. This created a stiffer sample after a period of time, so torque went up. A graph was produced by graphing torque vs. time. The sample was not destroyed because the sample was only being oscillated and not rotated continuously over a period of time. Since the rotor was straining the rubber, the resulted torque values were directly related to the shear

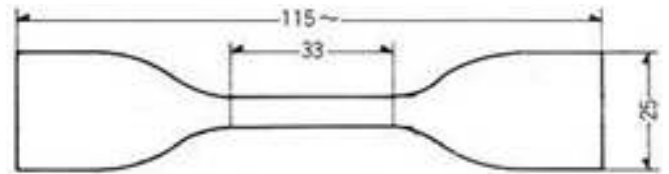
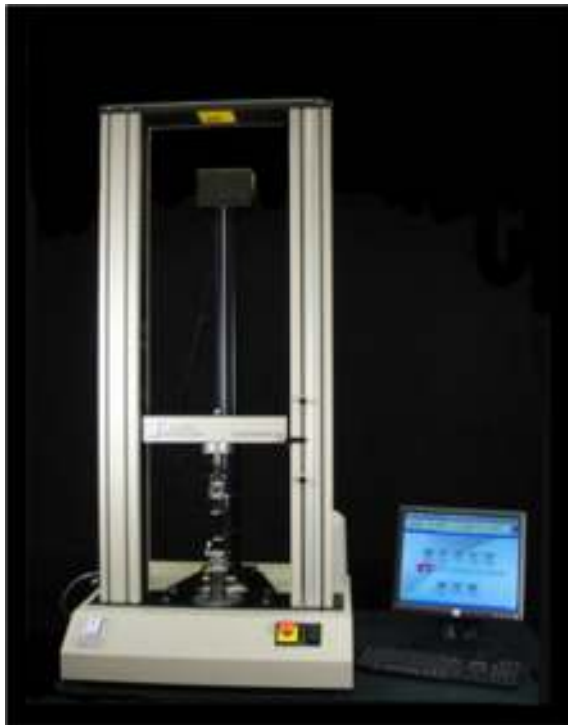
modulus of the sample.<sup>21</sup> Figure 3 contains a diagram of the ODR instrument, example of the rotor cavity, and a graph of a typical ODR sample.



**Figure 3 ODR Diagram, Rotor Cavity, and Graph of Sample**

Each compound had characteristic tensile measurements specific to the rubber compound. The tensile measurements were done with a tensometer. Results varied among the different compounds under study. The tensile tester was a way to quickly measure the quality of vulcanized rubber samples. The sample was pressed in an oven after being put into a mold, the specifications of this mold were 6 x 6 inch squares. The molds had a set thickness of approximately 0.075 inches, and depending on the amount of rubber placed in the mold the thickness of the sample may vary, consequently.<sup>22</sup> After the samples were “cured” they were ready to cut into the most commonly used tensile shape, the dumbbell. The term “cured” means that the compound had been exposed to a certain temperature for a length of time. This fully

cross-linked the sample so that it was properly tested by the instrument. Figure 4 contains an example of a commonly used tensometer and dumbbell used for tensile testing.



**Figure 4 Tensometer and Dumbbell Example**

The results for the tensometer followed the ASTM D412 testing parameters set for dumbbell pulls. ASTM D412 test methods cover procedures used to evaluate the tensile (tension) properties of vulcanized thermoset rubbers and thermoplastic elastomers. A few definitions below are listed below for clarity:

- Modulus: The amount of pull in pascals required to stretch the test piece to a given elongations. It expresses resistance to extension, or stiffness in the vulcanized rubber.
- Tensile: The force per unit of the original cross-sectional area which is applied at the time of rupture of the dumbbell test specimen. Tensile is recorded in pounds per square inch (psi)

- Elongation: The ability of rubber to stretch without breaking. This is typically expressed in percent.

Each company, has different testing standards for the compounds that was used in this study, so testing parameters and procedures varied.

The durometer was used directly on the compounds before the dumbbells of that compound were tested by the tensometer. Three dumbbells were aligned together and three consecutive readings were taken from a specific sample. The instrument used was a Shore A durometer, this was used for all of the compounds that were of interest. This property describes the rubber samples resistance to indentation.<sup>23</sup> The scale for this compound complied with ASTM D2240 parameters and had a scale of 0-100 units. Zero corresponded to a compound that is very soft, on the other hand, a Durometer of one hundred corresponded to a very stiff compound. Figure 5, below, contains an example of a Type A shore durometer.<sup>24</sup>



**Figure 5 Type A Shore Durometer**

The specific gravity of a compound refers to a comparison between its weight in water and air at a specific temperature. Typically, specific gravity is measured at approximately room temperature (25°C). In this research ASTM D297 standards were follow accordingly, so the

sample that was used for tensile slabs was cut into a 2-3 gram sample and weighed in air. The scale is tared and the sample was submersed into a 150 mL beaker containing distilled water. The weight was recorded and a calculation was performed. The next test that was performed on the rubber compounds was compression set.

Compression set was the property in rubber that was defined as the amount (%) by which a standard test piece failed to return to its original thickness after being subjected to a standard compressive load for a fixed period of time.<sup>25</sup> This information was important because it provided an approximation of real time rubber performance. For example, a weather strip in a vehicle is constantly being compressed and released due to the door being opened and closed. Compression set can help a chemist determine the best rubber compound for this application based on the results. Depending on the characteristic of the rubber compound, different times and temperatures were employed on the samples. There are several methods of measuring the compression set of rubber samples, but in this study Method B predominated. In method B, the sample is compressed to twenty-five percent it's original thickness for a set time and temperature. This was where buttons were cured under curing conditions that are described below Tables XXIV-XXVII. A button is a cured rubber piece that helps test the rubber compounds resistance to indentation. The buttons were between 0.470" and 0.510" thickness. The thickness was measured and the buttons were cured in a mold. Then they were placed between two metal plates and compressed to a thickness of 0.375".

The final part of this section dealt with all aged tensile results. Each of the compounds and natural alternatives were subjected to this test. This was a very useful study because it helped approximate the real life performance of the rubber compounds. With this in mind, it provided a comparison between the results of the control oil and natural oil alternatives. The study of aged

tensile was done in accordance with ASTM D573 standards. This study was done in an oven at a constant temperature for a certain period of time depending on the rubber compound. This exposed the rubber product to amplified conditions to test their reliability, deterioration rate, and overall performance. After the samples were exposed to the oven for a certain period of time the tensile samples were allowed to cool for at least nine hours in the lab.

## **Results**

Since each of the compounds under research had different testing conditions, each of those conditions were briefly described under Tables VIII-XI. Tables VIII-XI contained all of the Mooney viscometer results for each of the rubber compounds.

### **Table VIII Natural Rubber (Mooney Viscometer) Results**

Specimen	Mooney Scorch	
	ML (Mooney Units)	T5 (min)
Oil Used		
Control (790 T Liquid)	15.95	14.16
Palm	18.03	13.99
Soybean	19.28	14.60
Used Fryer	19.67	13.48
Canola	19.41	14.27
Safflower	18.51	14.38

### **Testing Parameters ASTM D1646**

- Preheat = 1 minute
- Test Temperature = 250 °F
- Test Duration = 30 minutes

### **Table IX PolyChloroprene (Mooney Viscometer) Results**

Specimen	Mooney Viscosity
Oil Used	ML (Mooney Units)
Control (Polycizer Butyl leate, Sundex 790 T liquid, and SI-69 liquid)	38.47
Palm	36.69
Soybean	35.57
Used Fryer	36.78
Canola	37.68
Safflower	38.44

### **Test Parameters ASTM D1646**

- Preheat = 1 minute
- Test Temperature = 212 °F
- Test Duration = 4 minutes

**Table X EPDM (Mooney Viscometer) Results**

Specimen	Mooney Viscosity
Oil Used	ML (Mooney Units)
Control (Sunpar 2280 Liquid)	17.69
Palm	16.39
Soybean	16.66
Used Fryer	17.17
Canola	15.91
Safflower	16.28

**Test Parameters ASTM D1646**

- Preheat = 1 minute
- Test Temperature = 250 °F
- Test Duration = 4 minutes

**Table XI Styrene Butadiene (Mooney Viscometer) Results**

Specimen	Mooney Scorch		Mooney Viscosity
	ML (Mooney Units)	T5 (min)	ML (Mooney Units)
Control (Calsol 8240 (2010) Liquid)	11.68	28.89	27.87
Palm	10.21	25.59	24.25
Soybean	10.46	28.16	25.24
Used Fryer	10.44	24.50	25.10
Canola	10.30	28.59	24.41
Safflower	10.95	28.13	26.23

**Test Parameters ASTM D1646**

**Mooney Scorch**

- Preheat = 1 minute
- Test Temperature = 250 °F
- Test Duration = 35 minutes

**Mooney Viscosity**

- Preheat = 1 minute
- Test Temperature = 212 °F
- Test Duration = 4 minutes



The instrumentation of the ODR was similar to the Mooney viscometer, and each compound had different testing specifications. Those specifications were listed below each compounds tabled results.

**Table XII Natural Rubber (ODR) Results**

Oil Used	ML (lb-in)	MH (lb – in)	t <sub>s</sub> 2 (min)	t <sub>c</sub> 50 (min)	t <sub>c</sub> 90 (min)
Control	4.71	48.25	1.27	1.81	3.40
Palm	5.49	45.76	1.17	1.68	3.05
Soybean	5.87	43.42	1.15	1.64	2.13
Used Fryer	5.95	45.14	1.19	1.68	3.39
Canola	5.97	44.06	1.17	1.67	2.20
Safflower	5.68	44.83	1.19	1.70	2.79

**Test Parameters ASTM D2084**

- Test Temperature = 350 °F
- Test Duration = 6 minutes
- Arc = 3°

**Table XIII PolyChloroprene Rubber (ODR) Results**

Oil Used	ML (lb-in)	MH (lb – in)	t <sub>s</sub> 2 (min)	t <sub>c</sub> 50 (min)	t <sub>c</sub> 90 (min)
Control	7.33	44.84	1.77	4.14	8.64
Palm	7.00	42.23	1.75	4.11	8.43
Soybean	7.12	40.35	1.78	4.13	8.44
Used Fryer	7.37	40.33	1.73	3.91	8.01
Canola	7.15	40.03	1.82	4.19	8.57
Safflower	7.51	41.12	1.84	4.20	8.47

**Test Parameters ASTM D2084**

- Test Temperature = 350 °F
- Test Duration = 12 minutes
- Arc = 3°

**Table XIV EPDM Rubber (ODR) Results**

Oil Used	ML (lb-in)	MH (lb – in)	t <sub>s</sub> 2 (min)	t <sub>c</sub> 50 (min)	t <sub>c</sub> 90 (min)
Control	5.72	70.46	0.92	2.58	4.25
Palm	4.85	49.14	0.87	2.34	3.86
Soybean	5.03	36.18	1.11	2.75	4.73
Used Fryer	5.32	40.37	1.00	2.69	4.63
Canola	4.93	41.97	0.99	2.63	4.54
Safflower	4.77	45.07	0.90	2.44	4.11

**Test Parameters ASTM D2084**

- Test Temperature = 350 °F
- Test Duration = 6 minutes
- Arc = 3°

**Table XV Styrene Butadiene Rubber (ODR) Results**

Oil Used	ML (lb-in)	MH (lb – in)	t <sub>c</sub> 2 (min)	t <sub>c</sub> 50 (min)	t <sub>c</sub> 90 (min)
Control	3.53	46.22	1.64	2.37	2.95
Palm	3.34	39.80	1.68	2.32	2.87
Soybean	3.38	34.35	1.64	2.23	2.83
Used Fryer	3.30	34.65	1.55	2.09	2.72
Canola	3.32	35.18	1.70	2.31	2.86
Safflower	3.52	37.56	1.69	2.32	2.93

**Test Parameters ASTM D2084**

- Test Temperature = 350 °F
- Test Duration = 4 minutes
- Arc = 3°

The next part of this section contained physical testing done with the tensometer. Each compound had characteristic tensile measurements specific to the rubber compound. So, results varied among the different compounds under study.

**Table XVI Natural Rubber Tensile Results**

Oil Used	100% Modulus (psi)	Tensile (psi)	Elongation (%)
Control	260.90	2673.90	563.80
Palm	242.10	2708.00	567.80
Soybean	237.20	2732.40	588.90
Used Fryer	231.00	2706.50	575.90
Canola	229.10	2645.30	600.80
Safflower	237.80	2626.10	585.80

**Test Parameters ASTM D412**

- Cure Temperature = 300 °F
- Cure Time = 45 minutes
- Tensile 100% Modulus = ≥ 160.00
- Tensile Strength = ≥ 2500.0
- Elongation = ≥ 400.00

**Table XVII Polychloroprene Rubber Tensile Results**

Oil Used	Tensile (psi)	Elongation (%)
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Control	2396.00	762.00
Palm	2425.00	750.00
Soybean	2485.00	736.00
Used Fryer	2395.00	762.00
Canola	2493.00	707.00
Safflower	2299.00	663.00

**Test Parameters ASTM D412**

- Cure Temperature = 350 °F
- Cure Time = 10 minutes
- Tensile Strength =  $\geq 1800.0$
- Elongation =  $\geq 400.00$

**Table XVIII EPDM Rubber Tensile Results**

Oil Used	Tensile (psi)	Elongation (%)
Control	1875.00	260.00
Palm	1697.00	366.00
Soybean	1656.00	553.00
Used Fryer	1724.00	530.00
Canola	1571.00	478.00
Safflower	1728.00	425.00

**Test Parameters ASTM D412**

- Cure Temperature = 350 °F
- Cure Time = 8 minutes
- Tensile Strength =  $\geq 1600.0$
- Elongation =  $\geq 300.0$

**Table XIX Styrene Butadiene Rubber Tensile Results**

Oil Used	300% Modulus (psi)	Tensile (psi)	Elongation (%)
Control	1200.00	2100.00	650.00
Palm	1158.00	2147.00	500.00
Soybean	959.00	1944.00	538.00
Used Fryer	1005.00	2023.00	538.00
Canola	1121.00	1989.00	494.00
Safflower	1118.00	1874.00	477.00

**Test Parameters ASTM D412**

- Cure Temperature = 350 °F
- Cure Time = 10 minutes
- Tensile 300% Modulus = (1050 – 1350)
- Tensile Strength =  $\geq 2000.0$
- Elongation =  $\geq 450.0$

The durometer and specific gravity were for compounding accuracy, because it was an easy way to validate that all components of the rubber compound were completely added during the mixing process. This was performed for all of the compounds under research and their test specifications are listed below Tables XX – XXIII.

**Table XX Natural Rubber (Durometer & Specific Gravity) Results**

Oil Used	Durometer	Weight in Air (grams)	Weight in H <sub>2</sub> O (grams)	Specific Gravity
Control	52	1.5642	0.2125	1.16
Palm	52	1.9444	0.2543	1.15
Soybean	52	1.7076	0.2167	1.14
Used Fryer	52	2.6567	0.3403	1.14
Canola	50	2.2245	0.2949	1.15
Safflower	51	2.2267	0.2547	1.13

**Durometer & Specific Gravity (ASTM D2240 & D297)**

- Durometer = (45-55)
- Specific Gravity = 1.200

**Table XXI Polychloroprene Rubber (Durometer & Specific Gravity) Results**

Oil Used	Durometer	Weight in Air (grams)	Weight in H <sub>2</sub> O (grams)	Specific Gravity
Control	47	2.7263	0.6113	1.29
Palm	48	3.1716	0.7284	1.30
Soybean	47	2.5638	0.5969	1.30
Used Fryer	47	1.9239	0.4445	1.30
Canola	46	2.2245	0.2949	1.29
Safflower	51	2.2267	0.2547	1.29

**Durometer & Specific Gravity (ASTM D2240 & D297)**

- Durometer = (40-50)
- Specific Gravity = (1.30)

**Table XXII EPDM Rubber (Durometer & Specific Gravity) Results**

Oil Used	Durometer	Weight in Air (grams)	Weight in H <sub>2</sub> O (grams)	Specific Gravity
Control	69	2.8229	0.2925	1.11
Palm	62	2.9731	0.3305	1.12
Soybean	60	2.7198	0.2995	1.12
Used Fryer	61	2.7787	0.3150	1.13
Canola	60	2.3599	0.2617	1.12
Safflower	63	2.3195	0.2638	1.13

**Durometer & Specific Gravity (ASTM D2240 & D297)**

- Durometer = (63-70)
- Specific Gravity = (1.090-1.150)

**Table XXIII Styrene Butadiene Rubber (Durometer & Specific Gravity) Results**

Oil Used	Durometer	Weight in Air (grams)	Weight in H <sub>2</sub> O (grams)	Specific Gravity
Control	57	2.7282	0.3197	1.13
Palm	56	2.7431	0.3104	1.13
Soybean	53	2.3429	0.2650	1.13
Used Fryer	54	2.4394	0.2838	1.13
Canola	54	2.4393	0.2838	1.13
Safflower	55	2.7307	0.3121	1.13

**Durometer & Specific Gravity (ASTM D2240 & D297)**

- Durometer = (52-60)
- Specific Gravity = (1.135-1.165)

Method B was used for the compounds listed in Tables XXIV-XXVII. The testing specifications for each compound depended on the nature of its constitute polymer. Testing parameters are placed below each table, in addition, all compression sets were done in accordance with ASTM D395 standards.<sup>26</sup>

**Table XXIV Natural Rubber (Compression Set) Results**

Control Oil		
Sample	1	2
Original Thickness (in.)	0.496	0.489
Final Thickness (in.)	0.472	0.466
Thickness % Change	19.8	20.2
Average Compression Set (%)	13.3	
Palm Oil		
Sample	1	2
Original Thickness (in.)	0.507	0.503
Final Thickness (in.)	0.479	0.474
Thickness % Change	21.2	22.7
Average Compression Set (%)	14.6	
Soybean Oil		
Sample	1	2
Original Thickness (in.)	0.506	0.506
Final Thickness (in.)	0.477	0.480

Thickness % Change	22.1	19.8
Average Compression Set (%)	13.9	
Used Fryer Oil		
Sample	1	2
Original Thickness (in.)	0.506	0.505
Final Thickness (in.)	0.474	0.474
Thickness % Change	24.4	23.8
Average Compression Set (%)	16.1	
Canola Oil		
Sample	1	2
Original Thickness (in.)	0.502	0.500
Final Thickness (in.)	0.472	0.472
Thickness % Change	23.6	22.4
Average Compression Set (%)	15.3	
Safflower Oil		
Sample	1	2
Original Thickness (in.)	0.505	0.506
Final Thickness (in.)	0.477	0.478
Thickness % Change	21.5	21.5
Average Compression Set (%)	14.3	

**Test Parameters ASTM D395**

- Cure Temperature: 300 °F
- Cure Time: 45 minutes
- Oven Temperature: 70 °C
- Time in Oven: 22 hours

**Table XXV Polychloroprene (Compression Set) Results**

Control Oil		
Sample	1	2
Original Thickness (in.)		0.486
Final Thickness (in.)	0.443	0.438
Thickness % Change	40.4	43.2
Average Compression Set (%)	41.8	
Palm Oil		
Sample	1	2
Original Thickness (in.)	0.500	0.497
Final Thickness (in.)	0.437	0.439
Thickness % Change	50.4	47.5
Average Compression Set (%)	48.9	
Soybean Oil		
Sample	1	2
Original Thickness (in.)	0.497	0.500
Final Thickness (in.)	0.436	0.437
Thickness % Change	50.0	50.4
Average Compression Set (%)	50.2	

Used Fryer Oil		
Sample	1	2
Original Thickness (in.)	0.499	0.491
Final Thickness (in.)	0.439	0.438
Thickness % Change	48.4	45.7
Average Compression Set (%)	47.0	
Canola Oil		
Sample	1	2
Original Thickness (in.)	0.492	0.500
Final Thickness (in.)	0.444	0.449
Thickness % Change	41.0	40.8
Average Compression Set (%)	40.9	
Safflower Oil		
Sample	1	2
Original Thickness (in.)	0.493	0.495
Final Thickness (in.)	0.442	0.444
Thickness % Change	43.2	42.5
Average Compression Set (%)	42.9	

### **Test Parameters ASTM D395**

- Cure Temperature: 350 °F
- Cure Time: 10 minutes
- Oven Temperature: 100 °C
- Time in Oven: 22 hours

### **Table XXVI EPDM Grade E (Compression Set) Results**

Control Oil		
Sample	1	2
Original Thickness (in.)	0.488	0.483
Final Thickness (in.)	0.477	0.471
Thickness % Change	9.70	11.1
Average Compression Set (%)	6.90	
Palm Oil		
Sample	1	2
Original Thickness (in.)	0.497	0.493
Final Thickness (in.)	0.468	0.464
Thickness % Change	23.8	24.6
Average Compression Set (%)	24.2	
Canola Oil		
Sample	1	2
Original Thickness (in.)	0.497	0.495
Final Thickness (in.)	0.452	0.455
Thickness % Change	36.9	33.3
Average Compression Set (%)	35.1	
Safflower Oil		
Sample	1	2

Original Thickness (in.)	0.487	0.492
Final Thickness (in.)	0.466	0.469
Thickness % Change	18.8	19.7
Average Compression Set (%)	12.8	

### **Test Parameters ASTM D395**

- Cure Temperature: 350 °F
- Cure Time: 8 minutes
- Oven Temperature: 100 °C
- Time in Oven: 22 hours

### **Table XXVII Styrene Butadiene Rubber (Compression Set) Results**

Control Oil		
Sample	1	2
Original Thickness (in.)	0.491	0.488
Final Thickness (in.)	0.477	0.474
Thickness % Change	12.1	12.4
Average Compression Set (%)	8.2	
Palm Oil		
Sample	1	2
Original Thickness (in.)	0.484	0.485
Final Thickness (in.)	0.463	0.465
Thickness % Change	19.3	18.2
Average Compression Set (%)	12.5	
Soybean Oil		
Sample	1	2
Original Thickness (in.)	0.495	0.485
Final Thickness (in.)	0.475	0.465
Thickness % Change	16.5	18.2
Average Compression Set (%)	11.6	
Used Fryer Oil		
Sample	1	2
Original Thickness (in.)	0.485	0.491
Final Thickness (in.)	0.469	0.474
Thickness % Change	14.5	14.7
Average Compression Set (%)	9.7	
Canola Oil		
Sample	1	2
Original Thickness (in.)	0.502	0.500
Final Thickness (in.)	0.472	0.472
Thickness % Change	23.6	22.4
Average Compression Set (%)	15.3	
Safflower Oil		
Sample	1	2
Original Thickness (in.)	0.494	0.483
Final Thickness (in.)	0.474	0.466
Thickness % Change	16.8	15.7



Average Compression Set (%)	10.8
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**Test Parameters ASTM D395**

- Cure Temperature: 350 °F
- Cure Time: 10 minutes
- Oven Temperature: 70 °C
- Time in Oven: 22 hours

The samples were then tested on the tensile tester and the results are listed in Tables XXVIII – XXXI. Testing parameters of each of the compounds were listed below each table, respectively.

**Table XXVIII Natural Rubber (Aged Tensile) Results**

Control Oil			
Sample	1	2	3
Thickness (in.)	0.076	0.076	0.071
Aged Tensile (PSI)	2657.4	2736.1	2812.1
Aged Elongation (%)	530.31	501.76	537.20
	Tensile		Elongation
Average % Change	2% increase		6% decrease
Aged Hardness	52	Hardness Change	0
Palm Oil			
Sample	1	2	3
Thickness (in.)	0.078	0.077	0.072
Aged Tensile (PSI)	2711.9	2841.0	2771.6
Aged Elongation (%)	548.31	534.15	544.31
	Tensile		Elongation
Average % Change	2% increase		4% decrease
Aged Hardness	52	Hardness Change	0
Soybean Oil			
Sample	1	2	3
Thickness (in.)	0.079	0.078	0.072
Aged Tensile (PSI)	2435.9	2692.5	3092.7
Aged Elongation (%)	584.52	550.71	582.30
	Tensile		Elongation
Average % Change	0.1% increase		1% decrease
Aged Hardness	52	Hardness Change	0
Used Fryer Oil			
Sample	1	2	3
Thickness (in.)	0.071	0.070	0.064
Aged Tensile (PSI)	2270.7	2439.2	2341.0
Aged Elongation (%)	543.42	547.63	554.97
	Tensile		Elongation

Average % Change	10% decrease		5% decrease
Aged Hardness	52	Hardness Changes	0
Canola Oil			
Sample	1	2	3
Thickness (in.)	0.072	0.064	0.072
Aged Tensile (PSI)	2627.5	2902.3	2432.2
Aged Elongation (%)	540.53	509.09	542.98
	Tensile		Elongation
Average % Change	0.7% decrease		10% decrease
Aged Hardness	50	Hardness Change	0
Safflower Oil			
Sample	1	2	3
Thickness (in.)	0.081	0.080	0.076
Aged Tensile (PSI)	2804.9	2646.2	2674.1
Aged % Elongation	569.86	526.11	556.63
	Tensile		Elongation
Average % Change	2% increase		5% decrease
Aged Hardness	51	Hardness Change	0

### **Test Parameters ASTM D395**

- Cure Temperature: 350 °F
- Cure Time: 10 minutes
- Oven Temperature: 70 °C
- Time in Oven: 70 hours

### **Table XXIX Polychloroprene Rubber (Aged Tensile) Results**

Control Oil			
Sample	1	2	3
Thickness (in.)	0.091	0.089	0.085
Aged Tensile (PSI)	2327.0	2489.0	2416.1
Aged Elongation (%)	524.98	537.46	550.31
	Tensile		Elongation
Average % Change	0.8% increase		29% decrease
Aged Hardness	47	Hardness Change	0
Palm Oil			
Sample	1	2	3
Thickness (in.)	0.086	0.081	0.085
Aged Tensile (PSI)	2454.2	2413.9	2488.5
Aged Elongation (%)	610.96	583.34	628.96
	Tensile		Elongation
Average % Change	1% increase		19% decrease
Aged Hardness	48	Hardness Change	0

Soybean Oil			
Sample	1	2	3
Thickness (in.)	0.077	0.080	0.088
Aged Tensile (PSI)	2501.7	2442.6	2435.1
Aged Elongation (%)	645.40	605.09	637.40
	Tensile		Elongation
Average % Change	2% decrease		13% decrease
Aged Hardness	47	Hardness Change	0
Used Fryer Oil			
Sample	1	2	3
Thickness (in.)	0.089	0.083	0.088
Aged Tensile (PSI)	2366.3	2517.7	2477.0
Aged Elongation (%)	646.73	639.38	673.39
	Tensile		Elongation
Average % Change	3% increase		15% decrease
Aged Hardness	47	Hardness Changes	0
Canola Oil			
Sample	1	2	3
Thickness (in.)	0.068	0.076	0.077
Aged Tensile (PSI)	2465.2	2468.3	2409.0
Aged Elongation (%)	624.51	616.91	636.73
	Tensile		Elongation
Average % Change	1% decrease		12% decrease
Aged Hardness	46	Hardness Change	0
Safflower Oil			
Sample	1	2	3
Thickness (in.)	0.083	0.089	0.090
Aged Tensile (PSI)	2420.8	2377.8	2396.4
Aged % Elongation	592.96	567.97	599.85
	Tensile		Elongation
Average % Change	4% increase		11% decrease
Aged Hardness	47	Hardness Change	0

### **Test Parameters ASTM D395**

- Cure Temperature: 350 °F
- Cure Time: 10 minutes
- Oven Temperature: 100 °C
- Time in Oven: 70 hours

### **Table XXX EPDM Grade E (Aged Tensile) Results**

Control Oil			
Sample	1	2	3
Thickness (in.)	0.074	0.080	0.079
Aged Tensile (PSI)	1978.3	2047.8	2031.5
Aged Elongation (%)	291.44	313.68	310.14
	Tensile		Elongation
Average % Change	8% increase		2% decrease

Aged Hardness	69	Hardness Change	0
Palm Oil			
Sample	1	2	3
Thickness (in.)	0.086	0.081	0.084
Aged Tensile (PSI)	1701.4	1631.5	1666.5
Aged Elongation (%)	503.21	466.29	484.75
	Tensile		Elongation
Average % Change	2% decrease		32% increase
Aged Hardness	62	Hardness Change	0
Canola Oil			
Sample	1	2	3
Thickness (in.)	0.085	0.086	0.079
Aged Tensile (PSI)	1824.4	1873.7	1765.2
Aged Elongation (%)	418.23	378.09	396.66
	Tensile		Elongation
Average % Change	9% increase		17% decrease
Aged Hardness	60	Hardness Change	0
Safflower Oil			
Sample	1	2	3
Thickness (in.)	0.076	0.080	0.087
Aged Tensile (PSI)	1707.0	1798.5	1766.6
Aged Elongation (%)	422.78	444.30	440.34
	Tensile		Elongation
Average % Change	2% increase		4% increase
Aged Hardness	63	Hardness Changes	0

### **Test Parameters ASTM D395**

- Cure Temperature: 350 °F
- Cure Time: 10 minutes
- Oven Temperature: 100 °C
- Time in Oven: 70 hours

### **Table XXXI Styrene Butadiene (Aged Tensile) Results**

Control Oil			
Sample	1	2	3
Thickness (in.)	0.075	0.073	0.083
Aged Tensile (PSI)	2119.9	2199.4	2046.1
Aged Elongation (%)	450.11	435.08	432.56
	Tensile		Elongation
Average % Change	0.9% increase		33% decrease
Aged Hardness	57	Hardness Change	0
Palm Oil			
Sample	1	2	3
Thickness (in.)	0.075	0.080	0.085
Aged Tensile (PSI)	2107.6	2162.3	2077.9

Aged Elongation (%)	504.54	508.85	507.43
	Tensile		Elongation
Average % Change	2% decrease		13% decrease
Aged Hardness	56	Hardness Change	0
Soybean Oil			
Sample	1	2	3
Thickness (in.)	0.082	0.080	0.075
Aged Tensile (PSI)	1944.6	1975.4	1974.1
Aged Elongation (%)	586.74	570.09	434.20
	Tensile		Elongation
Average % Change	0.03% increase		13% decrease
Aged Hardness	53	Hardness Change	0
Used Fryer Oil			
Sample	1	2	3
Thickness (in.)	0.076	0.077	0.085
Aged Tensile (PSI)	2008.6	2018.0	1996.6
Aged Elongation (%)	578.75	588.30	574.97
	Tensile		Elongation
Average % Change	0.7% decrease		8% increase
Aged Hardness	54	Hardness Changes	0
Canola Oil			
Sample	1	2	3
Thickness (in.)	0.081	0.080	0.076
Aged Tensile (PSI)	1860.5	1917.9	1992.0
Aged Elongation (%)	544.42	538.41	562.97
	Tensile		Elongation
Average % Change	4% decrease		10% increase
Aged Hardness	54	Hardness Change	0
Safflower Oil			
Sample	1	2	3
Thickness (in.)	0.079	0.082	0.087
Aged Tensile (PSI)	1884.2	1871.1	1867.2
Aged % Elongation	517.65	496.79	505.65
	Tensile		Elongation
Average % Change	0.2% decrease		6% increase
Aged Hardness	55	Hardness Change	0

### **Test Parameters ASTM D395**

- Cure Temperature: 350 °F
- Cure Time: 10 minutes
- Oven Temperature: 70 °C
- Time in Oven: 70 hours

### **Discussion**

Tables VIII-XI represent the results from the Mooney viscometer instrument used in this study. The significance of the results of the natural oils were compared relative to the petroleum

oil (control). For example, Table VIII contains the Mooney viscosometer results for the natural rubber compound. The control values for the Mooney scorch test were ML of 15.95 MU and T5 of 14.16 minutes. In order for the natural oils to be of novel interest, they must have the same or a longer T5 time relative to the control. Soybean oil had a ML of 19.28 MU and T5 of 14.60 minutes, consequently, this was found to be viable compound based on its T5 time. In contrast, the control had a much lower ML of 15.95 MU compared to an ML of 19.28 MU in soybean oil. This indicated that soybean oil would increase mechanical friction in the factory setting, so it is ultimately not a viable option. In Table IX, a Mooney viscosity test was performed on all of the samples and then all natural oils were compared to the control. The control had an ML of 38.47 MU, in contrast, soybean oil had a lower ML of 35.57 MU. Although Tables VIII-XI show promising results, in order for a natural oil to be viable as a replacement, all of the testing must be observed.

Tables XII-XV represent the results from the ODR instrument used in this study. The significance of these results follow the same protocol as the Mooney viscometer results. The ML, MH,  $t_{s2}$ , and  $t_{c90}$  were the most important readings that were taken from this instrument. They provided a picture on how the rubber compounds cross-linked or cured. The natural oils effected these readings differently than the petroleum oil (control). For example, Table XII displayed the control readings: ML 4.71 lb-in, MH 48.25 lb-in,  $t_{s2}$  1.27 minutes, and  $t_{c90}$  3.40 minutes. In comparison, palm oil had the most consistent results when compared to the control oil. The results for palm oil were: ML 5.49 lb-in, MH 45.76 lb-in,  $t_{s2}$  1.17 minutes, and  $t_{c90}$  3.05 minutes. The ML value for palm oil was slightly higher than the control, also the  $t_{c90}$  was slightly shorter in time due to a faster crosslinking rate. A faster crosslinking rate will increase the torque that is needed by the oscillating rotor to oscillate, which was seen in palm oil.

Tables XVI-XIX represent the results from the tensometer instrument used in this study. The companies that were included for this study each have their own testing parameters HEXPOL must meet in the lab. These specifications are below each of the tables, for example, Table XVI called for a 100% modulus (psi), tensile (psi), and elongation (%). The control yielded these results: 100% modulus 260.90 psi, 2673.90 psi, and 563.80 %. In order for any of the compounds to be considered as viable options they must have higher tensile and 100% modulus results. In Table XVI, it can be seen that palm and soybean oil exceed the control in both tensile and 100% modulus results. These are considered viable options, but compound viability depends on the overall results of the compounds containing natural oils.

Tables XX-XXIII contain all of the results collected for specific gravity and durometer on each of the rubber compounds. Table XX-XXIII will not be discussed because relative to the control all of the natural oil alternatives had similar or the same results. Tables XXIV-XXVII contain results of the compression sets performed on each of the rubber compounds. For example, Table XXIV contains the results for all of the natural rubber compression sets. The control had an average compression set of 13.3%, in comparison, soybean oil had a very similar result of 13.9%. The soybean oil may be considered as an alternative based on the similarities it had with the control. The rest of the results for compression set can be interpreted in this fashion.

Tables XXVIII-XXXI contain all of the results for the aged tensile tests performed on the rubber compounds. The best way to interpret these results is to compare the natural oils used with the control oil. For example, Table XXVIII contains results the control oil used in the selected natural rubber compound. The control oil has an average percent change of 2% in tensile and 6% in elongation. In comparison, palm oil had very similar results to the control with the

same average percent change in tensile and only a two percent difference in elongation. Palm oil may be of novel interest to replace the current petroleum oil (control).

Unfortunately, nitrile rubber compound did not mix with anything in the lab so this compound was not considered for further study. The rest of the rubber compounds mixed thoroughly and went through rigorous testing. In order to determine the best alternatives, all of these tests were necessary to provide a full picture on their performance.

The natural rubber compound had many studies performed on it, but the oils that performed the best, relative to the control, were: soybean, safflower, and palm oils. All of these oils were compared and contrasted with the control oil 790 T Liquid. This provided clarity on what exactly the results meant, also whether they were useful. The Mooney scorch results for the control were a  $M_L$  of 15.95 Mooney units and a T5 of 14.16 minutes. The  $M_L$  is the lowest point at which the Mooney viscometer instrument reads, then T5 refers to the time it takes for the compound to increase five Mooney units from the  $M_L$ . In comparison, these were the readings for the alternatives: soybean 19.28 & 14.60, palm 18.03 & 13.99, and safflower 18.51 & 14.38. All of the  $M_L$  readings for these alternatives produced a higher torque than the control oil. Although they produced a higher torque, the safflower and soybean oils had slightly longer T5 readings which implied that the process of curing in these compounds was slightly slower when in the presence of these oils. In contrast, the safflower oil was faster in reaching the T5 which meant a faster cure rate. These results were useful in contributing to the full picture because they allowed the rubber chemist to decide which oil is the better choice for the company's process.

The ODR results allowed the chemist more accuracy and precision in their measurements. The ODR instrument read several different things, but the most noteworthy in this study were the  $M_L$ ,  $M_H$ ,  $t_{s2}$ , and  $t_{c90}$ . The  $t_{s2}$  is how long the rubber compound took to



increase two units from the  $M_L$  reading, in addition, the  $t_{c90}$  was the time it took for the compound to reach 90% maximum torque. So, the  $M_L/M_H$  readings were in lb-in and  $t_s/t_{c90}$  readings were in minutes. This provided information about the compound's curing characteristics which was important in the factory when the customer produced their product. The results for the natural rubber compound can be found in Table XII.

The control had an  $M_L$  4.71,  $M_H$  48.25,  $t_s$  1.27, and  $t_{c90}$  3.40 which was typical of this compound. The readings for the substitutes were as listed: soybean 5.87, 43.42, 1.15, and 2.13, palm 5.49, 45.76, 1.17, and 3.05, safflower 5.68, 44.83, 1.19, and 2.79. All of the  $M_L$  readings for these substitute oils were higher for the compound, but only the  $t_{c90}$  will be discussed because it provided a better picture. The  $t_{c90}$  for the control was 3.05 minutes while the alternatives all had higher  $t_{c90}$  readings. This indicated that the time to 90% of the maximum torque was shorter with the alternatives than the control. Although, the readings were shorter this did not mean that these are not viable options. Palm and safflower had relatively close readings in terms of  $t_{c90}$  and if these performed well in other tests they may be viable options.

The specific gravity and durometer of these compounds were taken during this study. Each compound had a set range that it had to meet to be a viable option. All of the alternatives for the natural rubber compound met specifications, so they were not discussed. This trend continued for all of the other rubber compounds, unless a true deviation from this specification was found. So, the next set of results that were discussed in terms of the natural rubber compound was tensile/elongation, compression set, and aged tensile/elongation results.

For the natural rubber compound company, specifications had to be met for tensile and elongation of 2500.00 PSI and 400.00%. These results can be found in Table XVI, also this compound required a 100% modulus reading which was discussed too. The control had readings

of 2673.90, 563.60, and 260.90. In comparison, these were the results for the alternatives: soybean 2732.40, 588.90, and 237.20, palm 2709.00, 567.90, and 242.10, safflower 2626.10, 585.80, and 237.80. The first two alternatives listed had higher tensile and elongation readings than the original oil. The latter listed had a lower tensile, but a higher elongation, also the 100% modulus reading for all three alternatives were lower than the control. In this case, there was not much difference between these results, so compression set and aging results shed light on the differences between the alternatives and control.

The control results for compression set and aged tensile were both based on a percent of their original. The control had a compression set value of 13.3% and aged tensile results of 2% increase in tensile & 6% decrease in elongation. This meant that the control only compressed to 13.3% of its original thickness, in addition, after the specimens were aged and control all crosslinks were fully formed and over curing did not occur, so the compound was much stiffer. This resulted in a decrease in elongation and increase in tensile strength because tensile was the force per cross-sectional area. The alternatives compression set results were: soybean compression set: 13.9%, palm compression set: 14.6%, safflower compression set: 14.3%. The alternatives aged tensile results were: soybean 0.1% increase in tensile and 1% decrease in elongation, palm: 2% increase in tensile and 4% decrease in elongation, safflower: 2% increase in tensile and 6% decrease in elongation. In comparison, with the control oil compression set value soybean was the most relative with a value of 13.9%. The other two oils safflower and palm did not resist change in thickness under temperature as well as soybean. In contrast, the control oil did not match the alternatives in aged tensile results with higher decreases in elongation than all of the substitutes. Now that all of the results for this compound have been

discussed, the overall performance of these three alternatives were verified, but more testing and trials in the factory were necessary to qualify them to be implemented on the factory scale.

The polychloroprene compound had two promising alternatives based on their overall test results safflower and canola oils. The polychloroprene compound required three oils Polycizer Butyl leate, Sundex 790 T liquid, and SI-69 liquid. All of these oils were fully replaced by the natural oils, which were listed in the introduction. In comparison with the control, they performed consistently in the lab. The first set of results were the Mooney viscosity and ODR results. This compound did not require a Mooney Scorch test, so no results of this were discussed. The  $M_L$  in a Mooney Viscosity was taken after four minutes of the rubber being in the cavity. The control had a  $M_L$  of 38.47 MU, while the substitute's readings were: canola 37.68 and safflower 38.44 MU. Both of the natural oils produced lower  $M_L$  values which indicated that they produced softer polychloroprene compounds. The ODR readings for the control were:  $M_L$  7.33,  $M_H$  44.84,  $t_{s2}$  1.77, and  $t_{c90}$  8.44. The ODR for the natural oils were: canola  $M_L$  7.15,  $M_H$  40.03,  $t_{s2}$  1.82, and  $t_{c90}$  8.57, and safflower  $M_L$  7.51,  $M_H$  41.12,  $t_{s2}$  1.84, and  $t_{c90}$  8.47. Canola oil had fairly consistent readings with the control, but the  $t_{c90}$  reading for this compound was slightly longer than the control. Also, this compound had lower  $M_L$  and  $M_H$  readings which meant that the canola oil created a softer compound that produced less heat in the ODR instrument. In comparison, the control and safflower oil had consistent results, but the major difference was the higher  $M_L$  value for safflower oil. The tensile and elongation for this compound again had company specifications of 1800.00 psi (Tensile) and 400.00% (Elongation). The compounds must be above these specifications to be any use to the company which produced the specific part. The control compound had a tensile and elongation of 2396.00 psi and 762.00% which was above specification. The other two natural oils had tensile and

elongation readings of: canola 2493.00 PSI & 707.00% and safflower 2299.00 PSI & 663.00%. Although, canola oil had a higher tensile value it did not reach the same Elongation which meant a stiffer compound. The safflower oil had lower values, which indicated the same basic result. The next test that was performed on these compounds was the compression set. The change in thickness for the control was 41.8% from its original thickness value. The canola and safflower oils had compression set values of 40.9% and 42.9%, respectively. Also, the aged tensile results for the control were an increase of 0.8% in tensile and 29% decrease in elongation. In contrast, the canola had an increase in tensile of 1% and decrease in elongation of 12%. Safflower oil had an increase in tensile of 4% and decrease in elongation of 11%. Both of these compounds withstood heat more so than the control compound due to these percentages.

The EPDM Grade E compound and its different iterations were tested next. This compound was not tested for all of the compounds due to an interesting finding. The finding is termed as bleeding, which meant that the compound did not fully accept all of its oil due to incompatibility. The used fryer and soybean oils both bled after being cured as slabs for tensile testing. This was out of the scope of the research it will not be further discussed. This compound called for a Mooney Viscosity test and the  $M_L$  value for the control is 17.69. The only promising oil overall for this study was safflower, so its  $M_L$  value was 16.28. Although, we reached an  $M_L$  value for this iteration of the EPDM Grade E compound it's ODR did not pass specifications set by the company. The specifications are:  $M_L$  (5.00 – 8.00) lb-in,  $M_H$  (55.00 – 75.00) lb-in,  $t_{s2}$  (0.65 – 1.25) min, and  $t_{c90}$  (3.25 – 4.50) min. The results for the ODR run on the control were  $M_L$  5.72,  $M_H$  70.46,  $t_{s2}$  0.92, and  $t_{c90}$  4.25. In contrast, the safflower oil results were  $M_L$  4.77,  $M_H$  45.07,  $t_{s2}$  0.90, and  $t_{c90}$  4.11. The  $M_L$  and  $M_H$  of this compound were too low for the specifications so no other results were discussed. The mechanism for curing this compound was

by using peroxide there could be an interference from the implementing natural oils into this compound. This would require more research and was out of the scope of the current study.

The last compound in this study was the SBR compound and its iterations. There was no bleeding in this compound unlike in the EPDM Grade E compound. In this SBR compound though there were three natural oils that did not reach testing specification for tensile readings. These natural oils are used fryer, soybean, canola, and safflower oils. Due to this fact these oils were not looked at as viable compounds. The only oil that reached the specifications for tensile test specifications was palm oil. Consequently, the control and palm oils were the only compared and contrasted for the SBR compound. This compound required both a Mooney Viscosity and Mooney Scorch. So, the results for the control were listed in that order:  $M_L$  27.87 MU,  $M_L$  11.68 MU, and T5 28.89 minutes. There were specifications on the time allowed for this compound to T5 which was 20.00 to 30.00 minutes. Palm oil had values of:  $M_L$  26.23 MU,  $M_L$  10.21 MU, and T5 25.59 minutes. This value was slightly shorter than the control compound which indicated less processing safety. Instead of following the same trend as the other compounds let us evaluate the compound based on its overall result. In terms of aging and compression set the values for the palm oil did not hold up to the control. These values can be seen in Tables XXXI and XXVII.

### Conclusion

All of the compounds have been discussed in terms of their potential when tested against a control compound. Overall, the natural oils did not withstand heat as well as the petroleum based oils. So, due to the cheap price of petroleum oil and availability at commercial amounts, natural oils are not a viable option, at this time. If customers were to adopt some of the promising natural oils this may increase cost, but would make the company maintain a better environmental

standard. Green chemistry may find its way into rubber chemistry, but it may never fully overshadow the petroleum derived oils that are currently used in industry.

Consequently, the most noteworthy results from this study are included below in Tables XXXII – XXXV. The results of the most promising alternative oils are listed compared to the control, in addition, the best candidate is indicated by an asterisk in the following tables.

**Table XXXII Natural Rubber Candidate Oil Results**

<b>Results of Physical Testing (Natural Rubber)</b>						
	Mooney Scorch		ODR		Aged Tensile	Compression Set
Oil Used	M <sub>L</sub> (MU)	T5 (min.)	t <sub>s</sub> 2 (min.)	t <sub>c</sub> 90 (min.)	Average Percent Change (%)	Average Percent Change (%)
Control	15.95	14.16	1.27	3.40	2 Increase	13.3
Canola	19.41	14.27	1.17	2.20	0.7 Decrease	15.3
*Safflower	18.51	14.38	1.19	2.79	2 Increase	14.3
Soybean	19.28	14.60	1.15	2.13	0.1 Increase	13.9

**Table XXXIII Polychloroprene Candidate Oil Results**

<b>Results of Physical Testing (Polychloroprene)</b>					
	Mooney Viscosity	ODR		Aged Tensile	Compression Set
Oil Used	M <sub>L</sub> (MU)	t <sub>s</sub> 2 (min.)	t <sub>c</sub> 90 (min.)	Average Percent Change (%)	Average Percent Change (%)
Control	38.47	1.77	8.64	0.8 Increase	41.8
*Canola	37.68	1.82	8.57	1 Increase	40.9
Safflower	38.44	1.84	8.47	4 Increase	42.9
Palm	36.69	1.75	8.43	1 Increase	50.2

**Table XXXIV EPDM Candidate Oil Results**

Results of Physical Testing (EPDM)						
Oil Used	Mooney Scorch		ODR		Aged Tensile	Compression Set
	M <sub>L</sub> (MU)		t <sub>s2</sub> (min.)	t <sub>c90</sub> (min.)	Average Percent Change (%)	Average Percent Change (%)
Control	17.69		0.92	4.25	8 Increase	6.90
Palm	16.39		0.87	3.86	2 Decrease	24.2
Used Fryer	17.17		1.00	4.63	N/A	N/A
Soybean	16.66		1.11	4.73	N/A	N/A

**Table XXXV Styrene Butadiene Candidate Oil Results**

Results of Physical Testing (Styrene Butadiene)						
Oil Used	Mooney Scorch		ODR		Aged Tensile	Compression Set
	M <sub>L</sub> (MU)	T5 (min.)	t <sub>s2</sub> (min.)	t <sub>c90</sub> (min.)	Average Percent Change (%)	Average Percent Change (%)
Control	11.68	28.89	1.64	2.95	0.9 Increase	8.2
Canola	10.30	28.59	1.70	2.86	4 Decrease	15.3
*Safflower	10.95	28.13	1.69	2.93	0.2 Increase	10.8
Soybean	10.46	28.16	1.64	2.83	0.03 Increase	11.6

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