

Description

Title of the Invention

OIL-EXTENDED RUBBER, RUBBER COMPOSITION, AND METHOD FOR
MANUFACTURING THE OIL-EXTENDED RUBBER

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Technical Field

The present invention generally relates to an oil-extended rubber and a method for manufacturing the oil-extended rubber. The present invention also relates to a rubber composition, and a tire or a shoe sole containing the rubber composition.

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Background Art

There have been extensive researches for improving the physical properties of rubber compositions. Examples of such researches include the ones described in such patent documents as US2009/0176910, JP4335557, WO2008044722, KR2011073060, US2012/0065324, 15 US2008/0097023, US2011/0112215, and EP2072280. However, further studies for achieving the highest possible properties are still going on.

Purpose of the Invention

The purpose of this invention is to provide an oil-extended rubber and a rubber 20 composition which show improved physical properties.

Disclosure of Invention

The present inventors have unexpectedly found that the use of coconut oils with specific prescription can allow the rubber compositions containing the same to improve such physical 25 properties as abrasion resistance, and elastic properties including rebound and compression set.

According to first aspect of the present invention, there has been provided an oil-extended rubber comprising a vulcanizable rubber component, and a coconut oil with a free fatty acid content of 0.05% by mass or more.

According to second aspect of the present invention, there has been provided a rubber 30 composition comprising the oil-extended rubber according to the first aspect, and further comprising a diene-based rubber other than the vulcanizable rubber, and a rubber reinforcing agent.

According to third aspect of the present invention, there has been provided a rubber composition comprising a vulcanizable rubber component, a coconut oil with a free fatty acid

content of 0.05% by mass or more, a diene-based rubber other than the vulcanizable rubber, and a rubber reinforcing agent.

According to fourth aspect of the present invention, there has been provided a tire comprising the rubber composition according to the second aspect.

5 According to fifth aspect of the present invention, there has been provided a shoe sole comprising the rubber composition according to the second aspect.

According to sixth aspect of the present invention, there has been provided a method for manufacturing an oil-extended rubber, the method comprising a step of mixing a vulcanizable rubber component and a coconut oil with a free fatty acid content of 0.05% by mass or more.

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Brief Description of drawing

Figure 1 shows an example of the result of measurements on Payne Effect.

Figure 2 shows an example of the result of measurements on processability.

Figure 3 shows an example of the result of measurements on processability.

15 Figure 4 shows another example of the result of measurements on processability.

Figure 5 shows another example of the result of measurements on processability.

Description of Embodiments

As described above, an oil-extended rubber according to one aspect of the present invention contains a vulcanizable rubber component and a coconut oil with a free fatty acid content of 0.05% by mass or more.

Any vulcanizable rubber component can be used for the oil-extended rubber. Examples of the vulcanizable rubbers include polybutadiene and their derivatives. 1,4-cis-polybutadiene is preferably employed. Styrene-butadiene rubber (SBR) and Natural rubber (NR) can also be preferably employed.

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For instance, the vulcanizable rubber can be a polybutadiene rubber having the following properties:

The Mooney viscosity (ML1+4, 100°C) is preferably in the range of 29-90, and more preferably 40-85 and is further more preferably in the range of 43-80. A larger Mooney viscosity than the above range may deteriorate the mixing processability while a smaller one than the above range may lower the abrasion resistance undesirably and cold flow problem.

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The molecular weight distribution [Weight average molecular weight (Mw)/Number average molecular weight (Mn)] is in the range of 1.8-4.5, and more preferably in the range of

2.0-3.0. A larger molecular weight distribution than the above range may lower the abrasion resistance, while a smaller one than the above range may deteriorate the roll mill processability undesirably.

5 The weight average molecular weight (Mw) is preferably in the range of 400,000-1,200,000, and more preferably in the range of 500,000-1,000,000 and is further more preferably in the range of 550,000-850,000. A larger one than the above range may lower the roll mill processability, while a smaller one than the above range may lower the abrasion resistance undesirably.

10 The number average molecular weight (Mn) is preferably in the range of 120,000-600,000, and more preferably in the range of 150,000-500,000 and is further more preferably in the range of 200,000-400,000. A larger one than the above range may lower the roll mill processability, while a smaller one than the above range may lower the abrasion resistance undesirably.

15 The velocity dependence index (n-value) of the Mooney viscosity is in the range of 2.0-3.0, preferably in the range of 2.4-2.9, and more preferably in the range of 2.4-2.8. A smaller n-value than 2.3 worsens the ability incorporated into compound of filler, while a large one than 3.0 lower the rebound resilience undesirably.

20 The n-value index is determined from the degree of branching and the molecular weight distribution in the polybutadiene and is not correlated with the Mooney viscosity. A larger degree of branching or molecular weight distribution of the polybutadiene increases the n-value index, while a smaller degree of branching or molecular weight distribution decreases the n-value index.

25 The range of the n-value may be operated and changed in the following two stages because it is required to optimize the molecular weight distribution. First, in a butadiene polymerization stage, polybutadienes of several types with smaller n-values and different molecular weights are polymerized. Next, the polybutadienes of several types with different molecular weights are blended to widen the molecular weight distribution to adjust the n-value index of polybutadiene within an appropriate range. The n-value index in the polymerization stage can be adjusted with a mixed molar ratio of an organoaluminum compound serving as co-catalyst to water. In a word, an increased amount of water added to a certain amount of the organoaluminum compound reduces the mixed molar ratio, and as the mixed molar ratio becomes smaller, the n-value tends to become smaller. The mixed molar ratio of the organoaluminum compound serving as co-catalyst to water in the polymerizing stage is
30 preferably 2.0 or lower, and particularly preferably 1.0-1.8. A mixed molar ratio of 2.0 or higher

makes the n-value index too large while a mixed molar ratio lower than 1.0 may extremely lower the polymerization activity undesirably.

The 5% toluene solution viscosity (T_{cp}) and the Mooney viscosity (ML) have a ratio (T_{cp}/ML), which is preferably in the range of 2.0-4.0, and more preferably in the range of 2.5-3.0.

A larger T_{cp}/ML ratio than the above range increases the cold flow property of a rubber while a smaller one than the above range lowers the abrasion resistance undesirably.

The cis-1,4 content is preferably 95% or higher, more preferably 97% or higher, and particularly preferably 98% or higher. A lower cis-1,4 content than the above deteriorates the abrasion resistance undesirably.

The above polybutadiene can be produced in the presence of a cobalt-based catalyst. An example of the cobalt-based catalyst composition includes (A) a cobalt compound, (B) a halogen-containing organoaluminum compound, and (C) water.

The cobalt compound preferably employs salts and complexes of cobalt. Particularly preferable examples include cobalt salts such as cobalt chloride, cobalt bromide, cobalt nitrate, cobalt octylate (ethylhexanoate), cobalt naphthenate, cobalt acetate, and cobalt malonate; cobalt bisacetyl acetonate, and cobalt trisacetyl acetonate; acetoacetic acid ethyl ester cobalt; an organic basic complex such as a pyridine complex or picoline complex of a cobalt salt; and an ethyl alcohol complex.

Examples of the halogen-containing organoaluminum include trialkyl aluminum or dialkyl aluminum chloride, dialkyl aluminum bromide, alkyl aluminum sesquichloride, alkyl aluminum sesquibromide, and alkyl aluminum dichloride.

Examples of specific compounds include trialkyl aluminum such as trimethyl aluminum, triethyl aluminum, triisobutyl aluminum, trihexyl aluminum, trioctyl aluminum, and tridecyl aluminum.

Examples of the halogen-containing organoaluminum further include organoaluminum halides such as dialkyl aluminum chlorides such as dimethyl aluminum chloride and diethyl aluminum chloride, sesquiethyl aluminum chloride, and ethyl aluminum dichloride; and hydrogenated organoaluminum compound such as diethyl aluminum hydride, diisobutyl aluminum hydride, and sesquiethyl aluminum hydride. The organoaluminum compounds may be used in combination of two or more.

The molar ratio (B)/(A) between the component (A) and the component (B) is preferably 0.1-5000, and more preferably 1-2000.

The molar ratio (B)/(C) between the component (B) and the component (C) is preferably 0.7-5, more preferably 0.8-4, and particularly preferably 1-3.

Other than the butadiene monomer, they may contain a small amount of: conjugated dienes such as isoprene, 1,3-pentadiene, 2-ethyl-1,3-butadiene, 2,3-dimethylbutadiene, 2-methylpentadiene, 4-methylpentadiene, and 2,4-hexadiene; non-cyclic monoolefins such as ethylene, propylene, butene-1, butene-2, isobutene, pentene-1, 4-methylpentene-1, hexene-1, and octene-1; cyclic monoolefins such as cyclopentene, cyclohexene, and norbornene; and/or aromatic vinyl compounds such as styrene, and α -methylstyrene; and non-conjugated diolefins such as dicyclopentadiene, 5-ethylidene-2-norbornene, and 1,5-hexadiene.

Polymerization methods are not limited particularly. For example, bulk polymerization using a conjugated diene compound monomer such as 1,3-butadiene as a polymerization solvent, and solution polymerization may be applicable. Examples of the solvent in the solution polymerization include aromatic hydrocarbons such as toluene, benzene, and xylene; aliphatic hydrocarbons such as n-hexane, butane, heptane, and pentane; alicyclic hydrocarbons such as cyclopentane, and cyclohexane; olefin-based hydrocarbons such as the above olefin compounds, cis-2-butene, and trans-2-butene; hydrocarbon-based solvents such as mineral spirit, solvent naphtha, and kerosene; and halogenated hydrocarbon-based solvents such as methylene chloride.

Among the solvents above, toluene, cyclohexane, and a mixture of cis-2-butene with trans-2-butene are employed suitably.

Polymerization temperatures preferably fall within a range between -30°C and 150°C , and particularly preferably within a range between 30°C and 100°C . Polymerization periods of time preferably fall within a range between one minute and 12 hours, and particularly preferably within a range between five minutes and five hours.

After polymerization for a certain period of time, the inside of the polymerization vessel is depressurized if required, and then post treatments such as steps of cleaning and drying are taken.

The coconut oil used for the oil-extended rubber has a free fatty acid content of 0.05% by mass or more. The free fatty acid content is herein defined as a value measured by the test method according to AOAC (2012) 940.28. More specifically, each coconut oil sample is measured at room temperature. In this method, 5.0 gram of the each oil sample is prepared in Erlenmeyer flask. Then, 25 ml of isopropyl alcohol or ethanol is added and homogeneously mixed with oil sample. After that, 5-6 drops of phenolphthalein is added as a titration indicator. The oil solution is titrated with 0.1N NaOH solution until the color of mixture is changed to pink. The content of Free Fatty Acid (%FFA) is calculated as below:

Calculation

$$\%FFA = \frac{A \times B \times C}{D} \times 100$$

wherein: A = Titrated volume of NaOH solution (ml)

5 B = Concentration of NaOH solution (mol/liter)

C = Molecular weight of fatty acid (g/mol)

D = Weight of oil sample (g)

The present inventors have found that employing coconut oils having a free fatty acid
 10 content of 0.05% by mass or more can improve the physical properties of the rubber
 composition. As will be described in detail below, it has been found that the use of coconut oils
 having a free fatty acid content of 0.05% by mass or more in the rubber makes it possible to
 attain well balanced physical properties of the rubber composition.

The free fatty acid content is preferably 30% by mass or less, and is more preferably 18%
 15 by mass or less. When these are the cases, better balanced physical properties of the rubber
 composition can be attained.

Also, the free fatty acid content preferably is 0.1% by mass or more, more preferably
 falls in a range of 2 to 18% by mass, and much more preferably falls in a range of 3 to 12% by
 mass, and is further more preferably 5 to 9% by mass. Such conditions will improve physical
 20 properties of the rubber.

The iodine value of the coconut oil is arbitrary. However, it is preferable that the iodine
 value of the coconut oil is set as 10 or more. When this is the case, physical properties of the
 rubber composition can be improved further. Iodine value is herein defined as a value measured
 by the test method according to AOAC (2012) 993.20. More specifically, each coconut oil
 25 sample is measured at room temperature. 3.0 gram of the each oil sample is prepared in 500 ml
 Erlenmeyer flask (at least 2 blank determinations to run with each sample group are to be
 prepared as well). Then, 15 ml of cyclohexane-acetic acid solvent is added and completely
 dissolved with each oil sample. Then, Wijs solution is dispensed into flask containing test
 sample flask, stopper flask, and swirl to mix. Immediately timer is set for half an hour and flask
 30 is stored in dark at $25^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for duration of reaction. Then, sample flask is removed from dark
 environment. Then, 20 ml KI solution is added into sample flask and mixed. 150 ml of H_2O is
 added and the sample is gradually titrated with 0.1 mol/L standard $\text{Na}_2\text{S}_2\text{O}_3$ solution with
 constant and vigorous shaking or mechanical stirring. Titrating is continued until yellow color of

the sample has almost disappeared. 1-2 ml of starch indicator solution is added to flask and titrating is continued until blue color has just disappeared. Iodine value (IV) is calculated as below:

Calculation

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$$\text{Iodine value (IV)} = \frac{(B - S) \times M \times 12.69}{W}$$

wherein: B = Titration of blank (ml)

S = Titration of test sample (ml)

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M = Molarity of $\text{Na}_2\text{S}_2\text{O}_3$ solution

W = Weight of oil sample (g)

The content of the coconut oil is preferably ranging from 0.1 to 80 phr, and is more preferably ranging from 10 to 40 phr, and is further more preferably ranging from 21.5 to 37.5 phr. When these are the cases, viscosity of the oil-extended rubber can be optimized, making the productivity of the oil-extended rubber and the rubber composition become higher, and physical properties of the rubber composition can be improved and optimized further.

The coconut oil with a free fatty acid content of 0.05% by mass or more can be obtained as a crude coconut oil. Alternatively, such coconut oil can be obtained by refining a crude coconut oil and adding fatty acid such as lauric acid thereto. Also, such coconut oil can be obtained by heating up the crude oil, letting it cool down, separating it into clear part (refined coconut oil) and opaque part (oil with higher amount of fatty acids). When the crude oil is used, the cost for the oil-extended rubber or the rubber composition could be lower. When the refined oil with additional fatty acid or the oil obtained as the opaque part as described above is used, the physical properties of coconut oil and the oil-extended rubber or the rubber composition could be more stable. Refinement of the crude oil described above can be done either chemically or physically.

The oil-extended rubber can be manufactured, for example, by mixing a vulcanizable rubber component and a coconut oil with a free fatty acid content of 0.05% by mass or more.

The oil-extended rubber can be obtained by a solid-phase synthesis. Namely, the mixing step as described above can be performed without adding solvents. For example, the oil-extended rubber can be obtained as follows. Firstly, diene rubber is masticated by mixing equipment such as banbury mixer, kneader, two roll mills, or extruder (single screw or twin screw) around 1

minute at 90°C or less. Then, coconut oil is added with required amount of free fatty acid in masticated rubber for 3 minutes for well dispersion. In this way, coconut oil-extended polybutadiene rubber can be produced.

The oil-extended rubber can also be obtained by a liquid-phase synthesis. For example, the oil-extended rubber can be manufactured by (1) dissolving the vulcanizable rubber component in a solvent prior to performing the mixing step, and (2) using the dissolved vulcanizable rubber component in the mixing step. This method would make the mass production easier compared to the solid-phase synthesis as described earlier. Examples of the solvent for dissolving the vulcanizable rubber component include aliphatic alkanes such as n-hexane, cycloalkanes such as cyclohexane, and aromatic solvents such as toluene, benzene, and styrene. Among these solvents, cycloalkanes such as cyclohexane are most preferably employed as the solvent.

For example, the oil-extended rubber can be obtained by a liquid-phase synthesis as following procedure. 100 gram of 1,4-cis-polybutadiene rubber is dissolved in cyclohexane for 2-4 hours at room temperature. Coconut oil with required amount of free fatty acid is added into rubber solution. Coconut oil is homogenously mixed in rubber solution within 30 minutes. Coconut oil-extended polybutadiene rubber solution is dried in vacuum oven for 1 hour at 100 °C. In this way, coconut oil-extended polybutadiene rubber can be produced.

Furthermore, the oil-extended rubber can also be obtained by after 1,4-cis-polybutadiene rubber polymerization in the presence of a cobalt-based catalyst as mentioned above as in the following procedure. Polybutadiene polymerization is done following required specification of polymer properties such as Mooney Viscosity, Molecular weight, Molecular Weight Distribution (MWD), solution viscosity (T-cp). After the residence time, polymerization reaction is terminated by adding some amounts of water and antioxidant. Then, coconut oil with required amount of free fatty acid is added into rubber solution. Coconut oil is homogenously mixed in rubber solution within 30 minutes before de-solvent and drying process. Coconut oil-extended polybutadiene rubber solution is dried in vacuum oven for 1 hour at 100 °C. In this way, coconut oil-extended polybutadiene rubber can be produced.

A rubber composition according to one aspect of the present invention contains the oil-extended rubber as described above. Such rubber compositions have been found to show improved physical properties such as abrasion resistance and elastic properties including rebound and compression set. The content of the oil-extended rubber may be ranging from 1 to 100 phr, and preferably from 10 to 80 phr, and more preferably from 30 to 70 phr.

The rubber composition further contains a diene-based rubber other than the vulcanizable rubber. Examples of the diene-based rubber other than the vulcanizable rubber include butadiene rubber, natural rubber, isoprene rubber, styrene butadiene rubber, and a mixture thereof. Other examples thereof include high cis polybutadiene rubber, low cis polybutadiene rubber, emulsion-
5 polymerized styrene butadiene rubber or solution-polymerized styrene butadiene rubber (SBR), ethylene propylene diene rubber (EPDM), nitrile rubber (NBR), butyl rubber (IIR), chloroprene rubber (CR), and mixture thereof. Derivatives of these rubbers, for example, polybutadiene rubbers modified with tin compounds, or the above rubbers epoxy-modified, silane-modified, or maleic acid-modified may also be used solely or in combination of two or more. The content of
10 the diene-based rubber other than the vulcanizable rubber may be ranging from 1 to 100 phr, and preferably from 10 to 80 phr, and more preferably from 30 to 70 phr.

The rubber composition further contains a rubber reinforcing agent. Examples of the rubber reinforcing agent include silica, carbon black, and a mixture thereof. Other examples thereof include inorganic reinforcing agents such as various types of carbon black and white
15 carbon, carbon nanotube, clay, talcum, activated calcium carbonate, and ultrafine magnesium silicate; and organic reinforcing agents such as polyethylene resin, polypropylene resin, high styrene resin, phenol resin, lignin, modified melamine resin, cumarone indene resin, and petroleum resin. Particularly preferable examples include carbon black having a particle diameter of 90 nm or below and an amount of dibutyl phthalate (DBP) oil absorption number of
20 70 ml/100 g or more, for example, FEF, FF, GPF, SAF, ISAF, SRF, and HAF. The content of the rubber reinforcing agent may be ranging from 5 to 100 phr, and preferably from 10 to 80 phr, and more preferably from 25 to 75 phr. The rubber reinforcing agent most preferably contains silica and/or carbon black.

The rubber composition of the present invention may further contain compounding
25 ingredients kneaded therein, such as a vulcanizing agent, a vulcanization accelerator, an anti-oxidant, a filler, a rubber process oil, zinc oxide, and a stearic acid, if required, as generally used in the rubber industrial field.

Examples of the vulcanizing agent include publicly known vulcanizing agents, for example, sulfur, organic peroxides, resinous vulcanizing agents, and metal oxides such as a
30 magnesium oxide.

Examples of the vulcanization accelerator include publicly known vulcanization accelerators, for example, aldehydes, ammonias, amines, guanidines, thioureas, thiazoles, thiurams, dithiocarbamates, and xanthates.

Examples of the anti-oxidant include amine-ketone series, imidazole series, amine series, phenol series, sulfur series, and phosphorous series.

Examples of the filler include inorganic fillers such as calcium carbonate, basic magnesium carbonate, clay, litharge, diatomsaceous earth; and organic fillers such as reclaimed rubber and powdered rubber.

Examples of the rubber process oil include aromatic series, naphthenic series, and paraffinic series, either of which may be used.

The rubber composition can further contain a coconut oil in addition to the one having been already added to the oil-extended rubber. The coconut oil that can be additionally contained in the rubber composition may have a free fatty acid content of 0.05% by mass or more, or that of less than 0.05% by mass. By doing this, for example, the viscosity of the rubber composition can be properly adjusted.

In another embodiment, a rubber composition according to one aspect of the present invention contains a vulcanizable rubber component, a coconut oil with a free fatty acid content of 0.05% by mass or more, a diene-based rubber other than the vulcanizable rubber, and a rubber reinforcing agent. The rubber composition can further contain compounding ingredients, such as a vulcanizing agent, a vulcanization accelerator, an anti-oxidant, a filler, a rubber process oil, zinc oxide, and a stearic acid, if required, as generally used in the rubber industrial field. The specific examples of these components are the same as described above. Such embodiment can also result in an enhancement in such physical properties as abrasion resistance, and elastic properties including rebound and compression set.

The rubber composition described above can be used for tire application. The tire containing the rubber composition as described above has been found to show excellent performance in such properties as abrasion resistance, wet skid and ice skid resistance, and elastic properties including rebound and compression set.

The rubber composition described above can also be used for a shoe sole application. The shoe sole containing the rubber composition as described above has been found to show excellent performance in such properties as abrasion resistance, wet skid resistance, and elastic properties including rebound and compression set.

Examples

1. Preparation of coconut oil-extended polybutadiene rubber

The oil-extended rubber was obtained by a liquid-phase synthesis as follows. 100 gram of 1,4-cis-polybutadiene rubber was dissolved in cyclohexane for 2-4 hours at room temperature. Coconut oil with required amount of free fatty acid was added into rubber solution. Coconut oil

was homogenously mixed in rubber solution within 30 minutes. Coconut oil-extended polybutadiene rubber solution was dried in vacuum oven for 1 hour at 100 °C. In this way, coconut oil-extended polybutadiene rubber was produced.

2. Preparation of rubber composition

5 2-1. Non-Productive Mixing (Primary Compound)

During the non-productive mixing, all components except the vulcanizing agent and accelerators were mixed in the standard mixer such as a banbury mixer with initial temperature at 90 °C within 6 minutes mixing time. Firstly, all of mixtures of diene polymers were mixed in banbury mixer for 30 seconds. Then, half of filler especially silica and silane coupling agent were added in to mixer. At 1 minute and 30 seconds of mixing process, another half of filler and other rubber compound ingredients were added into mixer. Then, 2 minutes and 30 seconds later, ram of mixer chamber was opened up for cleaning residue filler trapped in the chamber. The mixing process had proceeded for 6 minutes. When mixing temperature reached 145 °C, the rotor speed of mixer had been reduced. The mixed compounds were rolled at preferred temperature range of 55-65 °C using a two roll mill mixer where distance to grind was set to 2 millimeters. The samples of the compound sheets obtained as above were subject to the Mooney viscosity measurement.

2-2. Productive Mixing (Secondary Compound)

The sheets of the primary compounds obtained from the aforementioned non-productive mixing were then subject to the mixing with vulcanizing agent, most preferably sulfur, and the vulcanizing accelerators by using two standard roller at preferred temperature range of 55-65 °C within 4 minutes. The rubber compounds from the productive mixing (secondary compound) have been pulled in sheets and the samples were then subject to the measurements of Mooney viscosity (ML1+4, 100 °C), curing time on a Moving Die Rheometer (MDR) at 160 °C.

25 3. Vulcanization and Properties of the Filler-Filled Vulcanizates

The secondary filler-filled rubber compounds obtained from the productive mixing were processed in the mold pressing at 160 °C according the curing time observed by a MDR as already mentioned. The rubber vulcanizates in the present invention in various forms of the specimens were then subject to the measurements of the viscoelastic property during the temperature sweep, tensile strength, hardness, specific gravity, tear resistance, rebound resilience, abrasion resistance, and compression set.

The viscoelastic property during the temperature sweep of the vulcanizates specimens in the present invention can directly relate to the results of the dynamic storage modulus E' , the dynamic loss modulus (E'') and the ratio of dynamic storage modulus and loss modulus, E''/E'

(tan delta). Generally, at low temperature region, the elastomeric or rubber materials with excellent viscoelastic property suitable for the application in tire treads should show the lower modulus at minus temperature (at higher than glass transition temperature), indicating the rubber state of the materials during being used at the snowing or icing environment, and the higher tan delta at minus temperature (at higher than glass transition temperature), indicating the better wet traction property during being used at the snowing or icing environment. Furthermore, the lower tan delta at high temperature (above room temperature) is preferred for the rubber materials with excellent viscoelastic property used in tire treads as this indicates the lower degree of hysteresis loss, hence the lower rolling resistance and lower fuel consumption.

4. Characterization Methods

(a) Microstructure of rubbers

Microstructure measurements were performed by FT-IR spectroscopy on a SHIMADZU-IRPrestige-21 using the standard KBR film and CS₂ solution methods.

(b) Molecular weight and molecular weight distribution of rubbers

Molecular weight and molecular weight distribution measurements were performed by Gel Permeation Chromatography (GPC) on SHIMADZU-CTO-20A GPC with two Shodex GPC KF-805L columns run in series at 40 °C column temperature in THF.

(c) Mooney Viscosity

Mooney viscosity (ML1+4, at 100 °C) measurement was performed in accordance with ASTM D1646 standard.

(d) Cure time of vulcanization

Cure time of vulcanization was determined from the time at 90 percent cured state of rubber compound (t₉₀), which was measured by Moving Die Rheometer (MDR) on an Alpha Technologies MDR2000 at 160 °C, constant frequency of 1.667 Hz and 0.5 degree of arc for torsional shear in accordance with ASTM D5289 standard. To be more specific, the following values were measured:

Min T (ML) = Minimum torque (unit dN.m)

Ts1 = Scorch time (unit min), time required for the increasing of 1 unit of torque from Minimum Torque. This number is an indication of the time required for the beginning of the process of crosslinking

Tc(10) = the time to 10 percent of torque increase or time corresponding to 10 percent curing of vulcanization

Max T (MH) = Maximum torque (unit dN.m)

(e) Viscoelastic property of rubber vulcanizates:

Dynamic temperature sweep analysis was performed on an EPLEXOR QC 25 (GABO, Germany) between -80 and 100 °C in tension mode at a constant frequency of 10 Hz, 1.0 % static strain and 0.1 % dynamic strain, heating rate 2 °C/minute.

(f) Abrasion resistance property of rubber vulcanizates:

5 Abrasion resistance was measured on the akron abrasion resistance machine according to the BS903 standard with standard weight 6lb. and sample angle of 15°.

(g) DIN Abrasion resistance property of rubber vulcanizates:

DIN Abrasion resistance measurement was performed in accordance with DIN:51536 standard.

10 (h) Rebound resilience property of rubber vulcanizates

Rebound resilience measurement was performed in accordance with BS903 standard part 22.

(i) Tensile property of rubber vulcanizates

15 Tensile measurement was performed in accordance with ASTM D412 standard with standard die cutter type C.

(j) Tear resistance property of rubber vulcanizates

Tear resistance measurement was performed in accordance with ASTM D624 standard.

(k) Hardness property of rubber vulcanizates:

20 Hardness measurement was performed in accordance with ASTM D2240 standard(shore A type).

(l) Compression set property of rubber vulcanizates

Compression set measurement was performed in accordance with ASTM D395.

5. Experiments

5-1. The effects of using specific oil-extended rubbers on various physical properties

25 5-1-1. Preparation and evaluation of oil-extended rubbers

The 1,4-cis-polybutadiene rubber used for the preparation is BR150L with Mooney viscosity of 52 as specified in Table 1 below as “P1.” The oil-extended rubbers P2, P3, P4, P5 and P6 were synthesized according to the method described above. Moreover, the oil-extended rubber with paraffinic oil (P7) was also prepared for comparison. The specification and Mooney viscosity of these polymers are summarized in Table 1 below.

Table 1

Polymer		P1	P2	P3	P4	P5	P6	P7
Oil	Type	-	Cocunut Oil	Cocunut Oil	Cocunut Oil	Cocunut Oil	Cocunut Oil	Parafinic oil
	Cocunut Oil FFA%	-	7%	7%	7%	2%	2%	-
	Iodine value (wijs)	NA	10.5	10.5	10.5	10.6	10.6	NA
	Content	-	21.5 phr	30 phr	40 phr	21.5 phr	30 phr	37.5 phr
Mooney viscosity	ML ₁₊₄ , 100°C	52	30.2	24.6	20.7	29.2	24.0	35

5-1-2. Preparation and evaluation of the rubber composition before vulcanization (secondary compound)

The secondary compounds were prepared according to the method described above and the recipe described in Table 2 below. In Table 2, Si69 means (Bis[3-(triethoxysilyl)propyl]tetrasulfide), St Acid means Stearic Acid, and AO.6C means (N-(1,3-Dimethylbutyl)-N'-phenyl-p-phenylenediamine).

Table 2

components	phr
S-SBR 1205	70
Oil-extended rubber	30
Silica (VN3) powder	75
Si69	6
Added oil	see Table 3
ZnO	3
St Acid	1
AO.6C	1

The specifications and physical properties of the secondary compounds are summarized in Table 3 below.

Table 3

Compound No.		C1 (Ref)	C2	C3	C4	C5	C6	C7 (Ref)	C8	C9
Polymer		P1	P6	P5	P3	P2	P4	P7	P5	P2
Additional Oil	Type	Sunthene 4240	Coconut FFA 2%	Coconut FFA 2%	Coconut FFA 7%	Coconut FFA 7%	Coconut FFA 7%	Sunthene 4240	Sunthene 4240	Sunthene 4240
	Iodine value (w/igs)	NA	10.6	10.6	10.5	10.5	10.5	NA	NA	NA
	phr	21.5	12.38	15	12.38	15	9.5	10.17	15	15
Mooney viscosity; 1st	ML ₁₊₄ , 100°C	68.5	62.4	61.8	55.3	52.9	58.9	75.2	76.4	72
Mooney viscosity; 2nd	ML ₁₊₄ , 100°C	60	45.2	44.2	40.6	39.2	43.1	58.3	59	55.7
Curing rate (160 °C)	Min. T (dN · m)	1.8	1.5	1.5	1.1	1.0	1.2	1.9	2.0	1.8
	Max. T (dN · m)	20.7	21.8	21.9	22.4	22.3	21.7	20.5	19.8	20.4
	Ts1 min.	1.2	1.3	1.2	1.5	1.5	2.0	0.5	1.4	1.4
	Tc(10) min.	2.4	3.1	2.6	3.3	3.4	3.4	2.2	2.4	3.0
	Tc(90) min.	7.56	7.51	7.35	8.16	8.41	7.51	7.24	7.1	7.3

5-1-3. Vulcanization and evaluation of the vulcanizates

The vulcanization was conducted according to the method described above and the recipe described in Table 4 below. In Table 4, CBS means (N-cyclohexyl-2-benzothiazole sulfenamide) and DPG means (Diphenylguanidine).

Table 4

components	phr
CBS	1.7
DPG	2
Sulfur	1.4

The physical properties of the vulcanizates are summarized in Table 5 below.

Table 5

Compound No.		C1 (Ref)	C2	C3	C4	C5	C6	C7 (Ref)	C8	C9
Hardness	Type A	76	74	74	74	74	74	75	76	76
Specific Gravity	-	1.188	1.186	1.188	1.187	1.187	1.187	1.181	1.186	1.186
100% Modulus	kg/cm ²	32	30	30	31	31	31	30	33	33
200% Modulus	kg/cm ²	62	58	58	62	60	61	60	65	65
300% Modulus	kg/cm ²	98	93	93	100	98	99	95	104	103
Tensile strength	kg/cm ²	143	152	149	155	149	152	148	142	140
Elongation	%	433	457	453	441	430	440	449	397	397
Tear resistance	kg/cm	57	59	57	59	59	59	58	60	61
Rebound	BS	%	40.9	43.8	42.9	44.3	44.3	41.4	42.9	42.9
Index vs Sunthene		100.0	107.1	104.9	108.3	108.3	108.3	101.2	104.8	104.8
Akron Abrasion	cc loss	0.058	0.045	0.043	0.040	0.039	0.040	0.056	0.049	0.052
Index vs Sunthene		100.0	128.9	134.9	145.0	148.7	145.0	103.6	118.4	111.5
DIN Abrasion	cc loss	116	115	117	120	125	121	126	N/A	N/A
Index vs Sunthene		100.0	100.9	99.1	96.7	92.8	95.9	92.1	N/A	N/A
Viscoelasticity										
-20°C										
E'	MPa	89.22	129.12	144.44	133.25	128.41	132.33	84.83	85.0	90.6
E''	MPa	9.66	16.62	17.94	16.78	16.19	16.42	8.79	10.4	10.4
E*	MPa	89.74	130.19	145.55	134.31	129.43	133.35	85.28	85.7	91.2
tand		0.108	0.129	0.124	0.126	0.126	0.124	0.104	0.123	0.115
Index vs Sunthene		100	119	115	116	117	115	96	113	106
0°C										
E'	MPa	68.80	81.78	91.77	83.06	84.60	87.02	67.88	61.8	68.7
E''	MPa	7.40	11.12	12.17	10.93	10.87	11.23	6.47	7.2	7.8
E*	MPa	69.20	82.53	92.57	83.78	85.29	87.74	68.19	62.2	69.2
tand		0.108	0.136	0.133	0.132	0.129	0.129	0.095	0.116	0.114
Index vs Sunthene		100	126	123	122	119	120	89	108	106
60°C										
E'	MPa	41.53	34.00	37.40	33.17	34.99	35.71	42.58	35.8	37.2
E''	MPa	5.16	4.87	5.47	5.05	5.11	5.04	5.02	4.4	4.7
E*	MPa	41.85	34.35	37.79	33.56	35.36	36.06	42.87	36.1	37.5
tand		0.124	0.143	0.146	0.152	0.146	0.141	0.118	0.124	0.127
Index vs Sunthene		100	87	85	82	85	88	105	100	98

As shown in Table 5, the physical properties are generally improved by employing coconut-oil extended rubbers.

5-2. The effects of adding specific coconut oils on various physical properties

5-2-1. Preparation and evaluation of the rubber composition before vulcanization (secondary compound)

The vulcanizable rubber used for the preparation is BR150L whose properties are summarized in Table 6 below.

Table 6

Name	BR150L
Mooney viscosity	ML ₁₊₄ , 100°C 44.4
T-cp	(cps) 108.6
Cis content	(%) 98.41

The secondary compounds were prepared according to the method described above and the recipe described in Table 7 below.

Table 7

components	phr
S-SBR 1205	70
BR150L	30
Silica (VN3) powder	75
Si69	6
Added oil	See Table 8
ZnO	3
St Acid	1
AO.6C	1

The specifications and physical properties of the secondary compounds are summarized in Table 8 below.

5 Table 8

Compound No.		C10 (Ref)	C11 (Ref)	C13	C14	C15 (Ref)	C16 (Ref)	C17 (Ref)	C18	C19	C20 (Ref)	C21 (Ref)	C22 (Ref)	C23	C24
Additional Oil	Type	Sunthene 4240	Soy bean	Coconut FFA 7%	Coconut FFA 2%	Sunthene 4240	Soy bean	Epoxidized Soy bean oil	Coconut FFA 7%	Coconut FFA 2%	Sunthene 4240	Soy bean	Epoxidized Soy bean oil	Coconut FFA 7%	Coconut FFA 2%
	Iodine value (w/ij)	NA	NA	10.5	10.6	NA	NA	1.87	10.5	10.6	NA	NA	1.87	10.5	10.6
	phr	21.5	21.5	21.5	21.5	15	15	15	15	15	10	10	10	10	10
Mooney viscosity, 1st	ML ₁₊₄ , 100°C	68.6	62.8	48.1	64.1	85.5	92.5	71.5	75.4	84.9	103.1	108.7	88	123.4	112.6
Mooney viscosity, 2nd	ML ₁₊₄ , 100°C	58.1	50.5	37.5	49	72.4	67.6	65.7	57	62.6	86.2	80.4	77.5	87.4	86.5
Curing rate (160 °C)	Min. T (dN · m)	1.6	2.0	1.1	3.0	2.1	2.4	2.4	1.9	2.5	2.4	2.7	2.7	3.6	2.9
	Max. T (dN · m)	20.5	18.0	23.2	22.9	22.7	21.4	18.2	26.4	25.8	25.9	24.7	22.7	31.2	26.4
	Ts1 min.	1.1	1.3	1.4	1.2	0.5	0.5	0.3	1.0	0.5	0.3	0.4	0.3	0.3	0.4
	Tc(10) min.	2.3	2.2	3.3	3.0	2.3	2.0	1.1	3.0	2.4	1.6	1.5	1.2	2.2	2.1
	Tc(90) min.	8.2	7.0	8.3	7.5	8.2	6.5	7.2	7.6	7.1	7.4	6.4	6.4	7.3	7.0

5-2-2. Vulcanization and evaluation of the vulcanizates

The vulcanization was conducted according to the method described above and the recipe described in Table 4 above. The physical properties of the vulcanizates are summarized in Table 9 below.

Table 9

Compound No.		C10 (Ref)	C11 (Ref)	C13	C14	C15 (Ref)	C16 (Ref)	C17 (Ref)	C18	C19	C20 (Ref)	C21 (Ref)	C22 (Ref)	C23	C24
Hardness	Type A	76-77	72-73	74-75	74-75	80-81	78-79	78-79	77-78	78-79	82-83	81-82	82-83	81-82	81-82
Specific Gravity	-	1.1879	1.1863	1.1875	1.1878	1.199	1.200	1.208	1.198	1.197	1.208	1.208	1.215	1.224	1.208
100% Modulus	kg/cm ²	29	23	29	30	34	31	30	34	34	37	37	37	41	40
200% Modulus	kg/cm ²	56	40	56	57	68	57	51	66	67	75	72	70	81	81
300% Modulus	kg/cm ²	89	63	92	91	111	92	80	106	107	118	114	110	128	132
Tensile strength	kg/cm ²	143	150	150	151	159	158	143	162	158	156	162	164	162	162
Elongation	%	460	655	468	466	402	490	528	439	426	392	420	450	376	359
Tear resistance	kg/cm	60	56	56	59	67	61	54	64	65	68	65	61	67	63
Rebound	BS	%	40.0	37.3	42.9	42.9	39.5	38.6	34.6	41.4	42.3	38.1	39.5	35.4	37.3
Index vs Sunthene		100	93	107	107	99	97	87	104	106	95	99	89	93	104
Akron Abrasion	cc loss	0.062	0.099	0.047	0.048	0.041	0.063	0.091	0.035	0.032	0.029	0.049	0.052	0.024	0.028
Index vs Sunthene		100	63	132	129	151	98	68	177	194	214	127	119	258	221
Viscoelasticity															
-20°C															
E'	MPa	88.6	88.6	151.3	150.2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
E''	MPa	9.7	10.7	18.2	18.8	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
E*	MPa	89.2	89.3	152.4	151.3	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
tand		0.109	0.121	0.120	0.125	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Index vs Sunthene		100	111	110	115	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
0°C															
E'	MPa	68.2	64.8	96.4	104.9	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
E''	MPa	7.4	7.6	12.3	13.8	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
E*	MPa	68.6	65.2	97.2	105.8	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
tand		0.109	0.117	0.128	0.131	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Index vs Sunthene		100	107	117	120	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
60°C															
E'	MPa	41.7	38.8	41.8	42.6	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
E''	MPa	5.2	5.3	5.9	5.8	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
E*	MPa	42.0	39.1	42.2	43.0	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
tand		0.124	0.136	0.141	0.136	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Index vs Sunthene		100	91	88	92	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

As shown in Table 9, the physical properties are generally improved by adding coconut oil as a component.

5 5-3. The effects of changing FFA content in the coconut oils on various physical properties

5-3-1. Preparation and evaluation of the rubber composition before vulcanization (secondary compound)

The vulcanizable rubber used for the preparation is BR150L whose properties are summarized in Table 6 above. The secondary compounds were prepared according to the method described above and the recipe described in Table 10 below.

Table 10

components	phr
S-SBR 1205	70
BR150L	30
Silica (VN3) powder	75
Si69	6
Added oil	21.5
ZnO	3
St Acid	1
AO.6C	1

The specifications and physical properties of the secondary compounds are summarized in Table 11 below. In this Table 11, “Refined coconut oil” had been prepared by refining crude coconut oil, and FFA content of the refined coconut oil is 0.07%.

Refined coconut oil was prepared as follows. That is, the crude coconut oil was heated up for some period of time and was allowed to cool. By doing so, the crude oil was separated into the clear part (edible oil with low melting point; refined coconut oil) and the opaque part (oil with higher amount of fatty acid with high melting point). This clear part was used as the “refined coconut oil.”

Also, the oils with “(RF)” mean the oils which are the opaque parts prepared by the refining process as described above which may have been adjusted the amount of free fatty acids in the desired range. On the other hand, the oils without “(RF)” mean that the coconut oil used in the example was a crude coconut oil which may have been adjusted the amount of free fatty acids in the desired range.

Table 11

Compound No.		C25 (Ref)	C26	C27	C28	C29	C30	C31	C32	C33	C34	C35	C36	C37	C38	C39
Additional Oil	Type	Sunthene4240	Refined coconut oil	FFA3%	FFA5%	FFA5%(RF)	FFA7%	FFA7%(RF)	FFA9%	FFA15%	FFA15%(RF)	FFA20%	FFA30%	FFA40%	FFA50%	FFA80%
	Iodine value (wjs)	NA	10.1	10.6	NA	NA	10.5	NA	NA	18.7	NA	NA	NA	NA	NA	NA
	phr	21.5	21.5	21.5	21.5	21.5	21.5	21.5	21.5	21.5	21.5	21.5	21.5	21.5	21.5	21.5
Mooney viscosity; 1st	ML ₁₊₄ , 100°C	63.8	54	49.2	46.3	47.5	44.6	44.6	43.9	40.3	39.9	38.3	40.8	43	52.6	78.1
Mooney viscosity; 2nd	ML ₁₊₄ , 100°C	54.8	44.1	40.3	37.7	39.8	36.8	36.6	35.8	33.7	33.2	32.1	34.3	37.9	44.9	63.0
Curing rate (160 °C)	Min. T (dN · m)	1.6	1.5	1.3	1.1	1.1	1.0	1.0	0.9	0.8	0.8	0.8	0.9	1.0	1.1	1.5
	Max. T (dN · m)	18.4	19.7	20.0	20.0	19.8	20.2	20.1	20.0	20.0	20.0	19.9	20.0	19.5	18.5	15.8
	Ts1 min.	1.2	0.5	1.2	1.3	1.3	1.4	1.5	2.1	2.5	2.5	2.5	2.5	2.5	2.4	2.0
	Tc(10) min.	2.3	2.2	2.5	3.1	3.1	3.1	3.2	3.3	3.5	3.5	3.5	3.4	3.2	3.1	2.1
	Tc(90) min.	8.11	8.00	8.07	8.24	8.08	8.03	8.30	8.17	8.12	8.17	8.01	7.40	7.06	6.28	5.13

5-3-2. Vulcanization and evaluation of the vulcanizates

The vulcanization was conducted according to the method described above and the recipe described in Table 4 above. The physical properties of the vulcanizates are summarized in Table 12 below.

Table 12

Compound No.			C25 (Ref)	C26	C27	C28	C29	C30	C31	C32	C33	C34	C35	C36	C37	C38	C39
Hardness	Type A		76	76	74-75	74-75	75	75	74	74-75	76	76	77	77	78	79	80
Specific Gravity	-		1.187	1.188	1.188	1.187	1.188	1.188	1.188	1.189	1.189	1.188	1.190	1.189	1.190	1.190	1.194
100% Modulus	kg/cm ²	32	30	31	31	31	31	31	31	32	37	36	39	41	40	40	39
200% Modulus	kg/cm ²	62	57	59	59	59	60	60	60	62	71	70	76	79	75	74	67
300% Modulus	kg/cm ²	99	91	95	95	97	98	97	97	101	113	112	119	120	114	111	100
Tensile strength	kg/cm ²	155	153	148	148	150	154	150	153	146	157	147	149	160	161	187	
Elongation	%	437	464	434	435	437	438	434	432	375	402	367	348	418	435	555	
Tear resistance	kg/cm	60	59	56	58	59	60	59	55	62	60	57	62	59	66	62	
Rebound	BS	%	40.3	42.9	41.4	44.3	42.3	43.8	42.9	43.8	42.9	42.9	41.4	41.4	39.5	37.3	32.0
Index vs Sunthene		100.0	106.4	102.8	110.0	105.0	108.6	106.4	108.6	106.4	106.4	102.8	102.8	97.9	92.5	79.5	
Akron Abrasion	cc loss	0.177	0.169	0.176	0.161	0.165	0.171	0.167	0.163	0.155	0.152	0.145	0.129	0.115	0.110	0.090	
Index vs Sunthene		100.0	104.7	100.6	109.9	107.3	103.5	106.0	108.6	114.2	116.4	122.1	137.2	153.9	160.9	196.7	
DIN Abrasion	cc loss	109	108	109	111	111	114	114	119	123	120	129	132	131	127	122	
Index vs Sunthene		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	
Compression Set	%	47.7	36.3	37.9	35.1	33.6	32.4	34.8	32.1	34.0	33.8	35.4	35.9	42.7	44.0	57.0	
Index vs Sunthene		100.0	131.4	125.9	135.9	142.0	147.2	137.1	148.6	140.3	141.1	134.7	132.9	111.7	108.4	83.7	
Viscoelasticity																	
-20°C																	
E'	MPa	58.0	96.8	102.2	99.7	98.4	82.2	96.5	94.5	97.2	92.8	94.5	N/A	96.8	N/A	122.3	
E''	MPa	9.0	15.8	16.6	16.7	16.0	13.8	16.3	15.8	15.8	15.5	15.7	N/A	15.4	N/A	18.7	
E*	MPa	58.7	98.0	103.5	101.1	99.7	83.4	97.9	95.8	98.5	94.1	95.8	N/A	98.0	N/A	123.7	
tand		0.155	0.163	0.163	0.168	0.163	0.168	0.169	0.167	0.162	0.167	0.167	N/A	0.159	N/A	0.153	
Index vs Sunthene		100	105	105	109	105	109	109	108	105	108	108	N/A	103	N/A	99	
0°C																	
E'	MPa	42.7	62.6	63.8	60.9	61.2	50.7	60.9	59.8	60.5	60.1	59.6	N/A	64.9	N/A	95.0	
E''	MPa	6.1	11.1	11.5	11.4	11.1	9.3	11.1	10.9	10.6	10.7	10.2	N/A	10.7	N/A	14.4	
E*	MPa	43.1	63.6	64.8	62.0	62.2	51.5	61.9	60.8	61.4	61.0	60.4	N/A	65.7	N/A	96.1	
tand		0.144	0.177	0.180	0.187	0.181	0.183	0.182	0.182	0.176	0.179	0.171	N/A	0.165	N/A	0.151	
Index vs Sunthene		100	123	125	130	126	127	126	127	122	124	119	N/A	115	N/A	105	
60°C																	
E'	MPa	23.1	21.1	23.4	21.4	22.0	17.7	21.9	21.4	22.7	22.4	23.5	N/A	24.9	N/A	34.4	
E''	MPa	3.7	3.9	4.4	3.9	4.1	3.2	4.0	4.0	4.0	3.9	4.0	N/A	4.3	N/A	7.2	
E*	MPa	23.4	21.4	23.8	21.7	22.3	18.0	22.2	21.8	23.1	22.7	23.8	N/A	25.3	N/A	35.2	
tand		0.160	0.186	0.187	0.181	0.184	0.180	0.183	0.187	0.177	0.175	0.171	N/A	0.172	N/A	0.208	
Index vs Sunthene		100	86	86	88	87	89	88	85	90	91	94	N/A	93	N/A	77	

As shown in Table 12, the physical properties are generally improved by adding coconut oil with various FFA content as a component.

- 5 5-4. The effects of changing the content of additional coconut oils on various physical properties
- 5-4-1. Preparation and evaluation of the rubber composition before vulcanization (secondary compound)

The vulcanizable rubber used for the preparation is BR150L whose properties are summarized in Table 6 above. The secondary compounds were prepared according to the method described above and the recipe described in Table 13 below.

Table 13

components	phr
S-SBR 1205	70
BR150L	30
Silica (VN3) powder	75
Si69	6
Added oil	See Table 14
ZnO	3
St Acid	1
AO.6C	1

The specifications and physical properties of the secondary compounds are summarized in Table 14 below.

Table 14

Compound No.		C40 (Ref)	C41	C42 (Ref)	C43	C44 (Ref)	C45	C46 (Ref)	C47	C48 (Ref)	C49	C50 (Ref)	C51	C52 (Ref)	C53	
Additional Oil	Type	Sunthene 4240	Coconut FFA7%	Sunthene 4240	Coconut FFA7%	Sunthene 4240	Coconut FFA7%	Sunthene 4240	Coconut FFA7%	Sunthene 4240	Coconut FFA7%	Sunthene 4240	Coconut FFA7%	Sunthene 4240	Coconut FFA7%	
	Iodine value (wijs)	NA	10.5	NA	10.5	NA	10.5	NA	10.5	NA	10.5	NA	10.5	NA	10.5	
	phr	10	10	15	15	21.5	21.5	30	30	37.5	37.5	50	50	80	80	
Mooney viscosity; 1st		ML ₁₊₄ ,100°C	99.3	86	74.8	66.3	60.9	43.3	43.8	29.1	35	19.2	23.9	12.2	12.6	5.4
Mooney viscosity; 2nd		ML ₁₊₄ ,100°C	82.6	69.8	63.8	53.8	52.5	36.0	39.1	24.7	32.7	16.3	22.4	10.3	11.8	5.7
Curing rate (160 °C)	Min. T (dN · m)	2.2	1.8	1.6	1.3	1.3	0.9	1.0	0.7	0.9	0.5	0.6	0.3	0.3	0.2	
	Max. T (dN · m)	22.6	24.0	20.9	21.8	17.3	19.4	14.0	16.4	12.4	14.8	9.0	11.2	4.8	6.7	
	Ts1 min.	1.0	1.2	0.6	1.3	1.3	2.0	2.2	2.6	2.4	3.3	3.3	4.3	5.2	6.1	
	Tc(10) min.	2.2	2.5	2.1	2.6	2.3	3.2	2.5	3.5	3.0	4.0	3.1	4.4	3.5	5.4	
	Tc(90) min.	7.21	7.30	7.29	7.45	8.01	8.16	8.32	8.43	8.47	9.13	9.32	9.58	10.54	12.20	

5

5-4-2. Vulcanization and evaluation of the vulcanizates

The vulcanization was conducted according to the method described above and the recipe described in Table 4 above. The physical properties of the vulcanizates are summarized in Table 15 below.

10 Table 15

Compound No.		C40 (Ref)	C41	C42 (Ref)	C43	C44 (Ref)	C45	C46 (Ref)	C47	C48 (Ref)	C49	C50 (Ref)	C51	C52 (Ref)	C53
Hardness	Type A	82	81	80	79	76	75	72	71	68	67	62	61	50	50
Specific Gravity	-	1.203	1.202	1.195	1.195	1.185	1.185	1.172	1.170	1.162	1.161	1.148	1.144	1.118	1.114
100% Modulus	kg/cm ²	40	40	35	36	30	30	24	27	22	24	17	20	12	13
200% Modulus	kg/cm ²	83	80	69	71	57	59	46	52	41	46	31	37	20	23
300% Modulus	kg/cm ²	134	130	110	115	90	98	73	84	63	75	47	58	29	34
Tensile strength	kg/cm ²	154	144	156	146	149	144	143	140	130	122	115	120	69	97
Elongation	%	341	328	408	370	462	414	539	462	590	457	659	554	689	697
Tear resistance	kg/cm	67	60	63	55	58	56	54	53	51	50	46	45	29	32
Rebound	BS	%	41.4	42.9	39.5	42.9	40.0	43.8	40.9	45.2	40.0	45.8	37.3	45.8	42.9
Index vs Sunthene		100	103.5	100	109	100	109	100	111	100	114	100	123	100	126
Akron Abrasion	cc loss	0.099	0.104	0.120	0.115	0.156	0.128	0.173	0.131	0.202	0.146	0.285	0.167	0.457	0.277
Index vs Sunthene		100	95.2	100	104	100	122	100	132	100	138	100	171	100	165
DIN Abrasion	cc loss	111	115	118	113	119	114	120	113	122	111	131	107	179	113
Index vs Sunthene		100	97	100	104	100	104	100	106	100	110	100	122	100	158
Compression Set	%	43.2	35.8	43.7	30.4	48.2	31.8	53.2	37.3	45.9	32.3	47.6	40.2	50.9	41.0
Index vs Sunthene		100	120.7	100	144	100	152	100	143	100	142	100	118	100	124
Viscoelasticity															
-20°C															
E'	MPa	75.9	88.3	70.4	85.6	59.9	90.4	43.1	100.9	42.6	93.6	N/A	N/A	19.6	147.3
E*	MPa	10.8	13.4	9.7	13.1	9.4	15.1	7.4	17.8	7.7	15.9	N/A	N/A	4.6	19.9
E*	MPa	76.7	89.3	71.0	86.6	60.7	91.7	43.8	102.5	43.3	94.9	N/A	N/A	20.1	148.7
tand		0.142	0.152	0.138	0.153	0.157	0.167	0.173	0.176	0.182	0.170	N/A	N/A	0.234	0.135
Index vs Sunthene		100	107	100	111	100	106	100	102	100	94	N/A	N/A	100	58
0°C															
E'	MPa	57.1	60.5	54.1	58.4	43.7	57.2	30.5	56.2	29.8	54.4	N/A	N/A	12.9	79.3
E*	MPa	7.9	9.5	7.2	9.2	6.8	10.5	5.2	11.7	5.4	10.9	N/A	N/A	2.8	13.7
E*	MPa	57.7	61.2	54.6	59.1	44.3	58.1	31.0	57.4	30.3	55.5	N/A	N/A	13.2	80.5
tand		0.138	0.157	0.133	0.158	0.155	0.183	0.170	0.208	0.180	0.201	N/A	N/A	0.217	0.173
Index vs Sunthene		100	114	100	119	100	118	100	122	100	112	N/A	N/A	100	80
60°C															
E'	MPa	32.1	30.9	29.6	25.2	24.4	19.9	16.9	15.4	16.5	11.3	N/A	N/A	6.5	4.7
E*	MPa	5.2	5.5	4.7	4.4	4.0	3.7	2.9	2.9	2.8	1.9	N/A	N/A	1.3	0.6
E*	MPa	32.5	31.4	30.0	25.6	24.7	20.3	17.2	15.6	16.8	11.4	N/A	N/A	6.6	4.7
tand		0.163	0.179	0.158	0.175	0.163	0.187	0.170	0.186	0.171	0.166	N/A	N/A	0.207	0.135
Index vs Sunthene		100	91	100	90	100	87	100	91	100	103	N/A	N/A	100	154

As shown in Table 15, the physical properties are generally improved by adding coconut oil with various amounts as a component.

5-5. The effects of changing the diene-based rubbers

5 5-5-1. Preparation and evaluation of the rubber composition before vulcanization (secondary compound)

The vulcanizable rubber used for the preparation is BR150L whose properties are summarized in Table 6 above. The secondary compounds were prepared according to the method described above and the recipe described in Table 16 below.

10 **Table 16**

components / phr	C54 (Ref)	C55	C56 (Ref)	C57
S-SBR 1205	70	70	-	-
E-SBR 1502	-	-	70	70
BR150L	30	30	30	30
Silica (VN3) powder	75	75	75	75
Si69	6	6	6	6
Added oil	21.5 (Sunthene oil 4240)	21.5 (Coconut FFA 7%)	21.5 (Sunthene oil 4240)	21.5 (Coconut FFA 7%)
ZnO	3	3	3	3
St Acid	1	1	1	1
AO.6C	1	1	1	1

The physical properties of the secondary compounds are summarized in Table 17 below.

Table 17

Compound No.		C54 (Ref)	C55	C56 (Ref)	C57
Additional Oil	Type	Sunthene 4240	Coconut FFA7%	Sunthene 4240	Coconut FFA7%
Mooney viscosity; 1st	ML ₁₊₄ , 100°C	59.9	45.4	67.9	56.1
Mooney viscosity; 2nd	ML ₁₊₄ , 100°C	51.2	36.4	56.9	48.5
Curing rate (160 °C)	Min. T (dN · m)	1.4	0.9	1.7	1.5
	Max. T (dN · m)	19.0	20.6	21.8	21.7
	Ts1 min.	1.2	1.5	1.5	1.5
	Tc(10) min.	2.2	2.6	3.0	2.5
	Tc(90) min.	7.28	7.41	8.34	7.22

5-5-2. Vulcanization and evaluation of the vulcanizates

- 5 The vulcanization was conducted according to the method described above and the recipe described in Table 4 above. The physical properties of the vulcanizates are summarized in Table 18 below.

Table 18

Compound No.			C54 (Ref)	C55	C56 (Ref)	C57
Hardness	Type A		77	78	69	70
Specific Gravity		-	1.186	1.185	1.187	1.187
100% Modulus	kg/cm ²		33	34	24	26
200% Modulus	kg/cm ²		65	68	59	62
300% Modulus	kg/cm ²		101	110	113	114
Tensile strength	kg/cm ²		150	154	161	154
Elongation	%		421	396	376	362
Tear resistance	kg/cm		62	62	69	61
Rebound	BS	%	32.0	41.4	50.0	52.0
Index vs Sunthene			100	129.4	100	104
Akron Abrasion	cc loss		0.142	0.112	0.151	0.134
Index vs Sunthene			100	126.8	100	113
Compression Set	%		44.2	31.2	21.6	23.9
Index vs Sunthene			100	142	100	90
DIN Abrasion	cc loss		115.0	109.0	106.0	103.0
Index vs Sunthene			100	105.5	100	102.9
Heat Build Up	ΔT	°C	40.0	40.0	25.0	21.0
	PS	%	35.5	26.8	10.2	6.4
Viscoelasticity						
-20°C						
E'	MPa		105.3	155.6	42.8	82.4
E''	MPa		14.0	22.5	10.3	15.9
E*	MPa		106.2	157.2	44.0	83.9
tand			0.133	0.145	0.241	0.193
Index vs Sunthene			100	109	100	80
0°C						
E'	MPa		81.0	89.1	30.2	47.8
E''	MPa		11.5	14.4	5.2	8.8
E*	MPa		81.8	90.3	30.6	48.6
tand			0.142	0.161	0.173	0.183
Index vs Sunthene			100	114	100	106
60°C						
E'	MPa		52.3	35.4	19.7	19.7
E''	MPa		7.4	6.5	2.4	2.4
E*	MPa		52.8	36.0	19.8	19.9
tand			0.142	0.184	0.121	0.123
Index vs Sunthene			100	77	100	99

As shown in Table 18, the physical properties are generally improved by adding coconut oil regardless of the type of the diene-based rubbers.

5-6. The effects of changing the additional oils

5 5-6-1. Preparation and evaluation of the rubber composition before vulcanization (secondary compound)

The vulcanizable rubber used for the preparation is BR150L whose properties are summarized in Table 6 above. The secondary compounds were prepared according to the method described above and the recipe described in Table 19 below.

10 Table 19

components / phr	C56 (Ref)	C57	C58 (Ref)	C59 (Ref)	C60 (Ref)	C61 (Ref)	C62 (Ref)	C63 (Ref)	C64 (Ref)	C65 (Ref)	C66 (Ref)
E-SBR 1502	70	70	70	70	70	70	70	70	70	70	70
BR150L	30	30	30	30	30	30	30	30	30	30	30
Silica (VN3) powder	75	75	75	75	75	75	75	75	75	75	75
Si69	6	6	6	6	6	6	6	6	6	6	6
Added oil	21.5 (Sunthene oil 4240)	21.5 (Coconut FFA 7%)	21.5 (Soybean oil)	21.5 (Palm oil)	21.5 (Corn oil)	21.5 (Rice Bran oil, Oryzanol 2500ppm)	21.5 (Rice Bran oil, Oryzanol 6000ppm)	21.5 (Sesame oil)	21.5 (Olive oil)	21.5 (Sun Flower oil)	21.5 (Canola oil)
ZnO	3	3	3	3	3	3	3	3	3	3	3
St Acid	1	1	1	1	1	1	1	1	1	1	1
AO.6C	1	1	1	1	1	1	1	1	1	1	1

The physical properties of the secondary compounds are summarized in Table 20 below.

Table 20

Compound No.		C56 (Ref)	C57	C58 (Ref)	C59 (Ref)	C60 (Ref)	C61 (Ref)	C62 (Ref)	C63 (Ref)	C64 (Ref)	C65 (Ref)	C66 (Ref)
Additional Oil	Type	Sunthene 4240	Coconut FFA7%	Soy-oil	Palm-oil	Corn-oil	Ri6000-Oil	Ri2500-Oil	Se-oil	Ol-oil	SunF-oil	Ca-oil
Mooney viscosity; 1st	ML ₁₊₄ , 100°C	67.9	56.1	61.4	58.9	61.1	59.6	61.4	61.8	62.9	65	64.3
Mooney viscosity; 2nd	ML ₁₊₄ , 100°C	56.9	48.5	53.1	55.8	53.8	52.5	53.9	53.3	55.0	54.8	55.3
Curing rate (160 °C)	Mn. T (dN · m)	1.7	1.5	1.7	1.6	1.6	1.6	1.6	1.7	1.7	1.8	1.8
	Max. T (dN · m)	21.8	21.7	19.4	20.6	19.9	20.2	20.0	19.7	20.7	19.5	19.9
	Ts1 min.	1.5	1.5	1.3	1.4	1.3	1.3	1.3	1.4	1.3	1.3	1.3
	Tc(10) min.	3.0	2.5	2.3	2.4	2.3	2.3	2.3	2.3	2.4	2.2	2.3
	Tc(90) min.	8.34	7.22	7.21	7.35	7.33	7.13	7.29	7.32	7.53	7.17	7.01

5 5-6-2. Vulcanization and evaluation of the vulcanizates

The vulcanization was conducted according to the method described above and the recipe described in Table 4 above. The physical properties of the vulcanizates are summarized in Table 21 below.

Table 21

Compound No.			C56 (Ref)	C57	C58 (Ref)	C59 (Ref)	C60 (Ref)	C61 (Ref)	C62 (Ref)	C63 (Ref)	C64 (Ref)	C65 (Ref)	C66 (Ref)
Hardness	Type A		69	70	67	68	66	68	68	67	68	67	67
Specific Gravity		-	1.187	1.187	1.187	1.186	1.188	1.187	1.188	1.189	1.188	1.189	1.188
100% Modulus		kg/cm ²	24	26	20	22	20	22	21	20	22	20	21
200% Modulus		kg/cm ²	59	62	43	51	46	48	46	45	50	43	47
300% Modulus		kg/cm ²	113	114	82	97	87	90	89	86	95	83	91
Tensile strength		kg/cm ²	161	154	217	172	187	189	185	179	183	190	199
Elongation		%	376	362	578	444	501	494	488	485	463	513	505
Tear resistance		kg/cm	69	61	70	66	69	67	62	69	65	65	65
Rebound	BS	%	50.0	52.0	48.9	49.8	49.8	48.9	50.4	47.3	48.9	48.9	51.3
Index vs Sunthene			100	104	98	100	100	98	101	95	98	98	103
Akron Abrasion		cc loss	0.151	0.134	0.188	0.141	0.170	0.168	0.142	0.165	0.140	0.164	0.162
Index vs Sunthene			100	113	80	107	89	90	106	92	108	92	93
Compression Set		%	21.6	23.9	25.7	25.6	25.5	23.3	23.4	25.4	26.5	25.4	25.1
Index vs Sunthene			100	90	84	84	85	93	92	85	82	85	86
DIN Abrasion		cc loss	106.0	103.0	92.0	98.0	96.0	100.0	98.0	96.0	101.0	96.0	97.0
Index vs Sunthene			100	102.9	115.2	108.2	110.4	106.0	108.2	110.4	105.0	110.4	109.3
Heat Build Up	ΔT	°C	25.0	21.0	33.0	26.0	32.0	28.0	29.0	31.0	27.0	32.0	31.0
	PS	%	10.2	6.4	14.8	9.6	13.3	11.6	11.5	12.4	10.7	14.8	13.2
Viscoelasticity													
-20°C													
E'		MPa	42.8	82.4	36.6	51.2	36.4	39.4	37.8				
E''		MPa	10.3	15.9	7.7	11.2	7.7	8.5	8.3				
E*		MPa	44.0	83.9	37.4	52.4	37.2	40.3	38.7				
tand			0.241	0.193	0.211	0.218	0.211	0.215	0.220				
Index vs Sunthene			100	80	88	91	88	89	91				
0°C													
E'		MPa	30.2	47.8	27.2	28.1	27.0	27.4	26.4				
E''		MPa	5.2	8.8	4.9	5.2	4.9	4.9	4.8				
E*		MPa	30.6	48.6	27.6	28.6	27.4	27.8	26.8				
tand			0.173	0.183	0.179	0.186	0.180	0.179	0.181				
Index vs Sunthene			100	106	104	107	104	104	105				
60°C													
E'		MPa	19.7	19.7	18.8	18.7	18.9	19.6	18.4				
E''		MPa	2.4	2.4	2.5	2.3	2.4	2.4	2.3				
E*		MPa	19.8	19.9	18.9	18.8	19.1	19.7	18.5				
tand			0.121	0.123	0.133	0.120	0.126	0.123	0.123				
Index vs Sunthene			100	99	91	101	96	99	98				

As shown in Table 21, the physical properties are generally improved by adding coconut oil compared to the cases where other additional oils are employed.

5-7. The effect of using coconut oil in polymer blends with standard carbon black compound

5 5-7-1. Non-Productive Mixing (Primary Compound)

During the non-productive mixing, all components except the vulcanizing agent and accelerators were mixed in the standard mixer such as a banbury mixer with initial temperature at 90 °C within 6 minutes mixing time. Firstly, half of diene polymers were put into banbury mixer, then, all of filler was added into mixer, after that another half of diene polymers were put into mixer. All of mixtures were mixed in banbury mixer for 3 minutes. Then, ram of mixer chamber was opened up for cleaning residue filler at 3 minute of mixing process. The mixing process had proceeded for 6 minutes or temperature reached 170 °C. The mixed compounds were rolled at preferred temperature range of 35-45 °C using the standard roller with nip

clearance of 6 millimeter. The samples of the compound sheets were left at room temperature for 1-24 hrs. Then, the compound samples were subject to the Mooney viscosity measurement.

The compositions are summarized in Tables 22 and 23 below, the former of which is described in phr and the latter of which is described in grams.

5 Table 22

	C67 (Ref)	C68	C69 (Ref)	C70	C71 (Ref)	C72	C73 (Ref)	C74
BR150L	100	100	50	50	50	50		
NR (ML = 70)			50	50			50	50
SBR1502					50	50	50	50
I R B #8	60	60	60	60	60	60	60	60
Sunthene#4240	15		15		15		15	
Coconut oil FFA7%		15		15		15		15
Z n O	3	3	3	3	3	3	3	3
stearic acid	2	2	2	2	2	2	2	2
	180	180	180	180	180	180	180	180
Compound	180	180	180	180	180	180	180	180
NS	0.9	0.9	0.9	0.9	0.9	0.9	0.9	0.9
sulfur	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
	182.4	182.4	182.4	182.4	182.4	182.4	182.4	182.4

Table 23

	C67 (Ref)	C68	C69 (Ref)	C70	C71 (Ref)	C72	C73 (Ref)	C74
BR150L	700	700	350	350	350	350	0	0
NR (ML = 70)	0	0	350	350	0	0	350	350
SBR1502	0	0	0	0	350	350	350	350
I R B #8	420	420	420	420	420	420	420	420
Sunthene#4240	105	0	105	0	105	0	105	0
Coconut oil FFA7%	0	105	0	105	0	105	0	105
Z n O	21	21	21	21	21	21	21	21
stearic acid	14	14	14	14	14	14	14	14
	1260	1260	1260	1260	1260	1260	1260	1260
Compound	1170	1170	1170	1170	1170	1170	1170	1170
NS	5.85	5.85	5.85	5.85	5.85	5.85	5.85	5.85
sulfur	12.19	12.19	12.19	12.19	12.19	12.19	12.19	12.19
	1188.0	1188.0	1188.0	1188.0	1188.0	1188.0	1188.0	1188.0

5-7-2. Productive Mixing (Secondary Compound)

- 10 The sheets of the primary compounds obtained from the aforementioned non-productive mixing were then subject to the mixing with vulcanizing agent, most preferably sulfur, and the vulcanizing accelerators by using the standard roll at preferred temperature range of 35-45 °C within 3 minutes. The rubber compounds from the productive mixing (secondary compound) have been pulled in sheets and the samples were then subject to the measurements of Mooney
- 15 viscosity (ML1+4,100 °C), curing time on a Moving Die Rheometer (MDR) at 160 °C.

The results are summarized in Table 24 below.

Table 24

Product name			C67 (Ref)	C68	C69 (Ref)	C70	C71 (Ref)	C72	C73 (Ref)	C74
		Oil Type	Sunthene	Coconut	Sunthene	Coconut	Sunthene	Coconut	Sunthene	Coconut
Mooney viscosity; 1st		ML ₁₊₄ , 100	75.4	67.9	60.1	57.4	64.3	61.4	48.7	47.0
Mooney viscosity; 2nd		ML ₁₊₄ , 100°C	65.0	58.6	46.3	44.9	55.9	53.1	38.3	36.1
Curing rate (160 °C)	Min. T	(dN · m)	2.7	2.6	2.0	2.0	2.2	2.1	1.6	1.5
	Max. T	(dN · m)	19.6	20.3	15.8	16.5	16.7	16.7	13.7	13.5
	Ts1	min.	3.0	2.6	3.0	2.5	4.2	4.2	3.5	3.4
	Tc(10)	min.	4.3	4.2	3.3	3.1	5.4	5.3	4.2	3.6
	Tc(90)	min.	11.00	11.12	7.08	6.58	16.34	16.24	11.21	10.45

5-7-3. Vulcanization and Properties of the Filler-Filled Vulcanizates

The secondary filler-filled rubber compounds obtained from the productive mixing were processed in the mold pressing at 145 °C and 35 minutes. The rubber vulcanizates in the present invention in various forms of the specimens were then subject to the measurements of the viscoelastic property during the temperature sweep, tensile strength, hardness, specific gravity, tear resistance, rebound resilience, abrasion resistance, and compression set.

The results are summarized in Table 25 below.

Table 25

Product name			C67 (Ref)	C68	C69 (Ref)	C70	C71 (Ref)	C72	C73 (Ref)	C74
		Oil Type	Sunthene	Coconut	Sunthene	Coconut	Sunthene	Coconut	Sunthene	Coconut
Hardness	Type A		64	64	62	62	59	59	61	60
Specific Gravity		-	1.114	1.115	1.119	1.118	1.127	1.126	1.132	1.131
100% Modulus		kg/cm ²	25	28	25	27	22	24	27	26
200% Modulus		kg/cm ²	59	67	64	70	51	57	68	69
300% Modulus		kg/cm ²	116	131	121	131	95	108	125	128
Tensile strength		kg/cm ²	182	179	238	221	209	190	236	228
Elongation		%	416	377	505	450	540	449	506	482
Tear resistance		kg/cm	66	61	60	58	64	58	59	54
Rebound	BS	%	64.6	64.6	56.8	60.2	50.4	52.9	45.8	48.2
vs Sunthene		Index	100	100	100	106	100	105	100	105
Akron Abrasion		cc loss	0.005	0.005	0.173	0.160	0.126	0.098	0.245	0.226
vs Sunthene		Index	100	100	100	108	100	129	100	108
Din Abrasion		cc loss	43	39	84	75	78	72	126	115
vs Sunthene		Index	100	110	100	112	100	108	100	110

As shown in Table 25, the physical properties are generally improved by using coconut oil compared to the cases where petroleum oil is used instead. It has also been found that such changes are more drastic when NR and/or SBR1502 were employed for the primary compound.

5-8. The effect of using coconut oil in polymer blends with silica compound

5-8-1. Non-Productive Mixing (Primary Compound)

During the non-productive mixing, all components except the vulcanizing agent and accelerators were mixed in the standard mixer such as a banbury mixer with initial temperature at 90 °C within 6 minutes mixing time. Firstly, all of mixtures of diene polymers were mixed in

banbury mixer for 30 seconds. Then, half of filler especially silica and silane coupling agent were added in to mixer. At 1 minute and 30 seconds of mixing process, another half of filler and other rubber compound ingredients were added into mixer. Then, ram of mixer chamber was opened up for cleaning residue filler. The mixing process had proceeded for 6 minutes. When
 5 mixing temperature reached 145 °C, the rotor speed of mixer had been reduced. The mixed compounds were rolled at preferred temperature range of 55-65 °C using the standard roller with nip clearance of 2 millimeter. The samples of the compound sheets were subject to the Mooney viscosity measurement.

The compositions are summarized is Tables 26 and 27 below, the former of which is
 10 described in phr and the latter of which is described in grams. The properties of various BRs are summarized in Table 28 below.

Table 26

	BR150L ML46		CBR(ML46) oil 21.5	CBR-(ML52) oil 21.5
	C75 (Ref)	C76	C77	C78
S-SBR 1205	70	70	70	70
BR150L	30	30	36.5	36.5
Silica (VN3) powder	75	75	75	75
Si69	6	6	6	6
Sunthene oil 4240	21.5		15.0	15.0
Coconut FFA 7%		21.5		
ZnO	3	3	3	3
St Acid	1	1	1	1
AO.6C	1	1	1	1
	207.5	207.5	207.45	207.45
CBS	1.7	1.7	1.7	1.7
DPG	2	2	2	2
Sulfur	1.4	1.4	1.4	1.4
Total	212.6	212.6	212.6	212.6

Table 27

			CBR(ML46)	CBR-(ML52)
	BR150L ML46		oil 21.5	oil 21.5
	C75 (Ref)	C76	C77	C78
S-SBR 1205	420	420	420	420
BR150L	180	180	218.7	218.7
Silica (VN3) powder	450	450	450	450
Si69	36	36	36	36
Sunthene oil 4240	129		90	90
Coconut FFA 7%		129		
ZnO	18	18	18	18
St Acid	6	6	6	6
AO.6C	6	6	6	6
	1245	1245	1244.7	1244.7
	1037.5	1037.5	1037.25	1037.25
CBS	8.5	8.5	8.5	8.5
DPG	10	10	10	10
Sulfur	7	7	7	7
Total	1063.0	1063.0	1062.8	1062.8

Table 28

Product name	Grade	CBR (Base ML46)	CBR (Base ML52)
	Load Coconut oil	21.5	21.5
CBR Mooney viscosity	ML ₁₊₄ ,100	23.0	26.5

5-8-2. Productive Mixing (Secondary Compound)

- 5 The sheets of the primary compounds obtained from the aforementioned non-productive mixing were then subject to the mixing with vulcanizing agent, most preferably sulfur, and the vulcanizing accelerators by using the standard roll at preferred temperature range of 60-70 °C within 4 minutes. The rubber compounds from the productive mixing (secondary compound) have been pulled in sheets and the samples were then subject to the measurements of Mooney viscosity (ML₁₊₄,100 °C), curing time on a Moving Die Rheometer (MDR) at 160 °C.
- 10

The results are summarized in Table 29 below.

Table 29

Product name		Oil Type	BR150L ML46		CBR(ML46)	CBR-(ML52)
		Oil phr	C75 (Ref)	C76	oil 21.5	oil 21.5
Mooney viscosity; 1st		ML ₁₊₄ ,100	57.9	46	60.1	58.5
Mooney viscosity; 2nd		ML ₁₊₄ ,100°C	53.4	39.1	51.8	50.2
Curing rate (160 °C)	Min. T	(dN · m)	1.6	1.1	1.5	1.4
	Max. T	(dN · m)	19.8	21.0	20.4	20.0
	Ts1	min.	1.0	2.2	1.1	1.2
	Tc(10)	min.	2.4	4.1	3.1	3.1
	Tc(90)	min.	8.41	9.10	8.43	8.43

5-8-3. Vulcanization and Properties of the Filler-Filled Vulcanizates

The secondary filler-filled rubber compounds obtained from the productive mixing were processed in the mold pressing at 150 °C according the curing time observed by a MDR as already mentioned (t90x2). The rubber vulcanizates in the present invention in various forms of the specimens were then subject to the measurements of the viscoelastic property during the temperature sweep, tensile strength, hardness, specific gravity, tear resistance, rebound resilience, abrasion resistance, and compression set.

The results are summarized in Table 30 below.

Table 30

Product name		Oil Type	BR150L	ML46	CBR(ML46)	CBR-(ML52)
		Oil phr	C75 (Ref)	C76	oil 21.5	oil 21.5
Hardness	Type A		76	75	76	76
Specific Gravity		-	1.189	1.189	1.189	1.189
100% Modulus		kg/cm ²	32	34	34	34
200% Modulus		kg/cm ²	63	66	66	67
300% Modulus		kg/cm ²	99	105	104	106
Tensile strength		kg/cm ²	141	134	140	141
Elongation		%	416	378	400	392
Tear resistance		kg/cm	61	66	66	64
Rebound	BS	%	41.4	44.3	42.9	42.9
vs Sunthene		Index	100.0	107.0	103.5	103.5
Lambourn Abrasion		(% vs sunthene)				
@ 20% slip rate			100.0	94.8	117.8	110.6
@ 40% slip rate			100.0	92.3	114.0	113.9
Heat Build UP:	ΔT	°C	25.3	22.4	24.9	23.6
	vs Sunthene	Index	100	113	101	107
	PS	%	37.9	29.7	35.6	37.4
Viscoelasticity						
-20°C						
E' (MPa)			105.4	151.8	105.4	104.0
E'' (MPa)			11.5	21.1	13.3	12.8
E* (MPa)			106.0	153.2	106.2	104.8
tand			0.109	0.139	0.126	0.123
Index (vs Sunthene)			100	127	115	113
0°C						
E' (MPa)			82.9	90.0	76.4	76.7
E'' (MPa)			9.2	13.0	9.5	9.2
E* (MPa)			83.4	90.9	77.0	77.3
tand			0.112	0.144	0.124	0.119
Index (vs Sunthene)			100	129	111	107
60°C						
E' (MPa)			49.5	36.6	43.1	43.1
E'' (MPa)			6.7	5.9	6.7	6.5
E* (MPa)			49.9	37.1	43.6	43.6
tand			0.136	0.162	0.156	0.1517
Index (vs Sunthene)			100	84	88	90

As shown in Table 30, the physical properties are generally improved by using coconut oil and coconut oil extended rubber compared to the cases where petroleum oil is used instead or the cases where coconut oil is not used.

5-8-4. Payne Effect

The Payne effect was measured after vulcanization of rubber specimens by using Alpha Technology RPA2000. The vulcanization process was done at 160°C, 30 minutes, then; temperature was decreased to 55°C. Vulcanized rubber specimens was measured the Payne effect under condition of temperature 55°C, frequency 1.667 Hz, strain 0.7-45%. The rubber compound characteristic such as storage modulus (G'), loss modulus (G''), and tan delta ($\tan\delta$) was measured and analyzed.

The results are shown in Figure 1. As shown in Figure 1, it has been found that C76-78 shows better performance than C75. It has also been found that C76 shows better performance than C77 and C78.

5-9. The effect of natural oil types on physical properties of SBR/BR blends silica compound

Preparation and analysis have been done in the same way as in the section 5-8. All the data are summarized in Tables 31, 32, 33 and 34 below.

Table 31

Oil types	C79 (Ref)	C80	C81 (Ref)	C82	C83 (Ref)	C84 (Ref)	C85 (Ref)	C86 (Ref)	C87 (Ref)	C88 (Ref)	C89 (Ref)	C90 (Ref)	C91 (Ref)
Various oil types recipe													
S-SBR1205	70	70											
E-SBR1502			70	70	70	70	70	70	70	70	70	70	70
BR150L	30	30	30	30	30	30	30	30	30	30	30	30	30
Silica (VN3) powder	75	75	75	75	75	75	75	75	75	75	75	75	75
Si69	6	6	6	6	6	6	6	6	6	6	6	6	6
Sunthene oil 4240	21.5		21.5										
Coconut FFA 7%		21.5		21.5									
Soybean oil					21.5								
Palm oil						21.5							
Corn oil							21.5						
Rice Bran oil (Oryzanol 2,500 ppm)								21.5					
Rice Bran oil (Oryzanol 6,000 ppm)									21.5				
Sesame oil										21.5			
Olive oil											21.5		
Sun Flower												21.5	
Canola oil													21.5
ZnO	3	3	3	3	3	3	3	3	3	3	3	3	3
St Acid	1	1	1	1	1	1	1	1	1	1	1	1	1
AO.6C	1	1	1	1	1	1	1	1	1	1	1	1	1
	207.5	207.5	207.5	207.5	207.5	207.5	207.5	207.5	207.5	207.5	207.5	207.5	207.5
CBS	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
DPG	2	2	2	2	2	2	2	2	2	2	2	2	2
Sulfur	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4
Total	212.6	212.6	212.6	212.6	212.6	212.6	212.6	212.6	212.6	212.6	212.6	212.6	212.6

Table 32

	C79 (Ref)	C80	C81 (Ref)	C82	C83 (Ref)	C84 (Ref)	C85 (Ref)	C86 (Ref)	C87 (Ref)	C88 (Ref)	C89 (Ref)	C90 (Ref)	C91 (Ref)
SSBR-1205	420	420											
E-SBR1502			420	420	420	420	420	420	420	420	420	420	420
BR150L	180	180	180	180	180	180	180	180	180	180	180	180	180
Silica (VN3) powder	450	450	450	450	450	450	450	450	450	450	450	450	450
Si69	36	36	36	36	36	36	36	36	36	36	36	36	36
Sunthene oil 4240	129		129										
Coconut FFA 7%		129		129									
Soybean oil					129								
Palm oil						129							
Corn oil							129						
Rice Bran oil (Oryzanol 2,500 ppm)								129					
Rice Bran oil (Oryzanol 6,000 ppm)									129				
Sesame oil										129			
Olive oil											129		
Sun Flower												129	
Canola oil													129
ZnO	18	18	18	18	18	18	18	18	18	18	18	18	18
St Acid	6	6	6	6	6	6	6	6	6	6	6	6	6
AO.6C	6	6	6	6	6	6	6	6	6	6	6	6	6
	1245	1245	1245	1245	1245	1245	1245	1245	1245	1245	1245	1245	1245
	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5
CBS	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5
DPG	10	10	10	10	10	10	10	10	10	10	10	10	10
Sulfur	7	7	7	7	7	7	7	7	7	7	7	7	7
Total	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0

Table 33

Product name			C79 (Ref)	C80	C81 (Ref)	C82	C83 (Ref)	C84 (Ref)	C85 (Ref)	C86 (Ref)	C87 (Ref)	C88 (Ref)	C89 (Ref)	C90 (Ref)	C91 (Ref)
		Oil Type	SunT-oil	Co-oil	SunT-oil	Co-oil	Soy-oil	Palm-oil	Corn-oil	Ri6000-Oil	Ri2500-Oil	Se-oil	Ol-oil	SunF-oil	Ca-oil
Mooney viscosity; 1st		ML ₁₊₄ , 100	59.9	45.4	67.9	56.1	61.4	58.9	61.1	59.6	61.4	61.8	62.9	65.0	64.3
Mooney viscosity; 2nd		ML ₁₊₄ , 100°C	51.2	36.4	56.9	48.5	53.1	55.8	53.8	52.5	53.9	53.3	55.0	54.8	55.3
Curing rate	Min. T	(dN • m)	1.4	0.9	1.7	1.5	1.7	1.6	1.6	1.6	1.6	1.7	1.7	1.8	1.8
(180 °C)	Max. T	(dN • m)	19.0	20.6	21.8	21.7	19.4	20.6	19.9	20.2	20.0	19.7	20.7	19.5	19.9
	Ts1	min.	1.2	1.5	1.5	1.5	1.3	1.4	1.3	1.3	1.3	1.4	1.3	1.3	1.3
	Tα(10)	min.	2.2	2.6	3.0	2.5	2.3	2.4	2.3	2.3	2.3	2.3	2.4	2.2	2.3
	Tα(90)	min.	7.28	7.41	8.34	7.22	7.21	7.35	7.33	7.13	7.29	7.32	7.53	7.17	7.01

Table 34

Product name			C79 (Ref)	C80	C81 (Ref)	C82	C83 (Ref)	C84 (Ref)	C85 (Ref)	C86 (Ref)	C87 (Ref)	C88 (Ref)	C89 (Ref)	C90 (Ref)	C91 (Ref)
		Oil Type	SunT-oil	Co-oil	SunT-oil	Co-oil	Soy-oil	Palm-oil	Corn-oil	Ri6000-Oil	Ri2500-Oil	Se-oil	Ol-oil	SunF-oil	Ca-oil
Hardness	Type A		77	78	69	70	67	68	66	68	68	67	68	67	67
Specific Gravity		-	1.186	1.185	1.187	1.187	1.187	1.186	1.188	1.187	1.188	1.189	1.188	1.189	1.188
100% Modulus		kg/cm ²	33	34	24	26	20	22	20	22	21	20	22	20	21
200% Modulus		kg/cm ²	65	68	59	62	43	51	46	48	46	45	50	43	47
300% Modulus		kg/cm ²	101	110	113	114	82	97	87	90	89	86	95	83	91
Tensile strength		kg/cm ²	150	154	161	154	217	172	187	189	185	179	183	190	199
Elongation		%	421	396	376	362	578	444	501	494	488	485	463	513	505
Tear resistance		kg/cm	62	62	69	61	70	66	69	67	62	69	65	65	65
Rebound	BS	%	32.0	41.4	50.0	52.0	48.9	49.8	49.8	48.9	50.4	47.3	48.9	48.9	51.3
vs Sunthene			100.0	129.4	100.0	104.0	97.8	99.6	99.6	97.8	100.8	94.6	97.8	97.8	102.6
Akron Abrasion		cc loss	0.142	0.112	0.151	0.134	0.188	0.141	0.170	0.168	0.142	0.165	0.140	0.164	0.162
vs Sunthene			100.0	126.8	100.0	112.7	80.3	107.1	88.8	89.9	106.3	91.5	107.9	92.1	93.2
Compression Set		%	44.2	31.2	21.6	23.9	25.7	25.6	25.5	23.3	23.4	25.4	26.5	25.4	25.1
vs Sunthene			100.0	141.7	100.0	90.4	84.0	84.4	84.7	92.7	92.3	85.0	81.5	85.0	86.1
Din Abrasion		cc loss	115	109	106	103	92	98	96	100	98	96	101	96	97
Cut Growth Resistance	Stroke	Kcycle	40.0	20.0	45.0	76.7	65.0	33.3	50.7	43.3	38.3	52.0	26.7	52.5	32.5
(ASTM D813 : 2-15 mm)															
57 mm		Index	100	50	100	170	144	74	113	96	85	116	59	117	72
Heat Build Up	DT	(°C)	40.0	40.0	25.0	21.0	33.0	26.0	32.0	28.0	29.0	31.0	27.0	32.0	31.0
	PS	%	35.5	26.8	10.2	6.4	14.8	9.6	13.3	11.6	11.5	12.4	10.7	14.8	13.2
Viscoelasticity															
-20°C															
E' (MPa)			105.3	155.6	42.8	82.4	36.6	51.2	36.4	39.4	37.8	N/A	N/A	N/A	N/A
E'' (MPa)			14.0	22.5	10.3	15.9	7.7	11.2	7.7	8.5	8.3	N/A	N/A	N/A	N/A
E* (MPa)			106.2	157.2	44.0	83.9	37.4	52.4	37.2	40.3	38.7	N/A	N/A	N/A	N/A
tand			0.133	0.145	0.241	0.193	0.211	0.218	0.211	0.215	0.220	N/A	N/A	N/A	N/A
Index (vs Sunthene)			100	109	100	80	88	91	88	89	91	N/A	N/A	N/A	N/A
0°C															
E' (MPa)			81.0	89.1	30.2	47.8	27.2	28.1	27.0	27.4	26.4	N/A	N/A	N/A	N/A
E'' (MPa)			11.5	14.4	5.2	8.8	4.9	5.2	4.9	4.9	4.8	N/A	N/A	N/A	N/A
E* (MPa)			81.79	90.26	30.62	48.57	27.63	28.56	27.40	27.84	26.82	N/A	N/A	N/A	N/A
tand			0.142	0.161	0.173	0.183	0.179	0.186	0.180	0.179	0.181	N/A	N/A	N/A	N/A
Index (vs Sunthene)			100	114	100	106	104	107	104	104	105	N/A	N/A	N/A	N/A
60°C															
E' (MPa)			52.3	35.4	19.7	19.7	18.8	18.7	18.9	19.6	18.4	N/A	N/A	N/A	N/A
E'' (MPa)			7.4	6.5	2.4	2.4	2.5	2.3	2.4	2.4	2.3	N/A	N/A	N/A	N/A
E* (MPa)			52.8	36.0	19.8	19.9	18.9	18.8	19.1	19.7	18.5	N/A	N/A	N/A	N/A
tand			0.142	0.184	0.121	0.123	0.133	0.120	0.126	0.123	0.123	N/A	N/A	N/A	N/A
Index (vs Sunthene)			100	77	100	99	91	101	96	99	98	N/A	N/A	N/A	N/A

5

As shown in Table 34, the physical properties are generally improved by using coconut oil compared to the cases where other types of oils are used instead. In addition, S-SBR1205

compound system showed the better improvement of physical properties than E-SBR compound system.

5-10. The comparison of physical properties between coconut oil extended BRs (CBRs), which have various ML viscosity of BR matrix and oil contents

5 Preparation and analysis have been done in the same way as in the section 5-8. All the data are summarized in Tables 35, 36, 37, 38 and 39 below.

Table 35

	C92 (Ref)	C93	C94	C95	C96	C97	C98	C99	C100
E-SBR1502	70	70	70	70	70	70	70	70	70
BR150L	30	30							
BR150L High ML (ML=52)			30						
High ML matrix (ML = 70)				30					
150L-oil extended 21.5					36.45				
150L High ML-oil extended 21.5						36.45			
150L High ML-oil extended 30							39		
High ML matrix (ML=70)-oil extended 21.5								36.45	
High ML matrix (ML=70)-oil extended 30									39
Silica (VN3) powder	75	75	75	75	75	75	75	75	75
Si69	6	6	6	6	6	6	6	6	6
Sunthene oil 4240	21.5				15.05	15.05	12.5	15.05	12.5
Coconut FFA 7%		21.5	21.5	21.5					
ZnO	3	3	3	3	3	3	3	3	3
St Acid	1	1	1	1	1	1	1	1	1
AO.6C	1	1	1	1	1	1	1	1	1
	207.5	207.5	207.5	207.5	207.5	207.5	207.5	207.5	207.5
CBS	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
DPG	2	2	2	2	2	2	2	2	2
Sulfur	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4
Total	212.6	212.6	212.6	212.6	212.6	212.6	212.6	212.6	212.6

Table 36

	C92 (Ref)	C93	C94	C95	C96	C97	C98	C99	C100
E-SBR1502	420	420	420	420	420	420	420	420	420
BR150L	180	180							
BR150L High ML (ML=52)			180						
High ML matrix (ML = 70)				180					
150L-oil extended 21.5					218.7				
150L High ML-oil extended 21.5						218.7			
150L High ML-oil extended 30							234		
High ML matrix (ML=70)-oil extended 21.5								218.7	
High ML matrix (ML=70)-oil extended 30									234
Silica (VN3) powder	450	450	450	450	450	450	450	450	450
Si69	36	36	36	36	36	36	36	36	36
Sunthene oil 4240	129				90.3	90.3	75	90.3	75
Coconut FFA 7%		129	129	129					
ZnO	18	18	18	18	18	18	18	18	18
St Acid	6	6	6	6	6	6	6	6	6
AO.6C	6	6	6	6	6	6	6	6	6
	1245	1245	1245	1245	1245	1245	1245	1245	1245
	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5
CBS	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5
DPG	10	10	10	10	10	10	10	10	10
Sulfur	7	7	7	7	7	7	7	7	7
Total	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0

Table 37

Product name	Grade	BR150L		BR150L(ML=52)			High ML matrix (ML=70)		
	Coconut oil(phr)	0	21.5	0	21.5	30	0	21.5	30
Mooney viscosity	ML ₁₊₄ , 100	42.6	22.6	50.0	25.6	20.0	70.0	38.0	31.5

Table 38

Product name			C92 (Ref)	C93	C94	C95	C96	C97	C98	C99	C100
		Oil Type	Sunthene	Coconut	Coconut	Coconut	Coconut	Coconut	Coconut	Coconut	Coconut
Mooney viscosity; 1st		ML ₁₊₄ , 100	63.6	50.9	50.1	58.0	57.9	56.8	57.5	65.4	64.7
Mooney viscosity; 2nd		ML ₁₊₄ , 100°C	54.7	44.8	43.6	52.1	50.6	49.9	50.3	56.9	56.6
Curing rate (160 °C)	Min. T	(dN · m)	1.7	1.4	1.4	1.5	1.5	1.4	1.5	1.7	1.7
	Max. T	(dN · m)	21.7	21.2	21.3	21.5	21.4	21.2	21.0	21.3	21.3
	Ts1	min.	2.0	2.2	2.2	2.1	2.0	2.1	2.1	1.6	2.0
	Tc(10)	min.	3.2	3.2	3.2	3.2	3.1	3.1	3.1	3.1	3.1
	Tc(90)	min.	9.11	8.36	8.50	8.50	8.52	8.53	8.44	8.50	8.50

Table 39

Product name			C92 (Ref)	C93	C94	C95	C96	C97	C98	C99	C100
		Oil Type	Sunthene	Coconut	Coconut	Coconut	Coconut	Coconut	Coconut	Coconut	Coconut
Hardness	Type A		67	69	69	69	68	66	67	67	67
Specific Gravity		-	1.187	1.188	1.189	1.189	1.188	1.186	1.187	1.188	1.188
100% Modulus		kg/cm ²	24	25	26	27	24	22	25	24	27
200% Modulus		kg/cm ²	61	61	65	67	60	55	61	62	66
300% Modulus		kg/cm ²	118	116	121	124	114	107	115	120	124
Tensile strength		kg/cm ²	183	177	162	168	158	170	158	170	175
Elongation		%	401	406	369	372	378	404	370	380	382
Tear resistance		kg/cm	68	63	65	65	67	63	62	62	68
Rebound	BS	%	52.9	52.9	52.0	53.6	53.6	52.0	53.6	52.9	54.5
vs Sunthene		Index	100.0	100.0	98.3	101.3	101.3	98.2	101.2	100.0	103.0
Akron Abrasion		cc loss	0.156	0.130	0.141	0.131	0.140	0.137	0.144	0.130	0.136
vs Sunthene		Index	100.0	120.0	110.6	119.1	111.4	113.9	108.3	120.0	114.7
Compression Set		%	22.0	22.6	21.1	19.3	19.1	19.6	20.9	18.6	16.9
vs Sunthene		Index	100.0	97.3	104.3	114.0	115.2	112.2	105.3	118.3	130.2

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As shown in Table 39, the physical properties are generally improved by using coconut oil compared to the case where other type of oil is used instead. In addition, the higher ML viscosity of BR matrix of coconut oil extended BR showed the better improvement of physical properties than the lower ML viscosity of BR matrix.

- 10 5-11. The comparison of physical properties between coconut oil extended BR (CBR) and other types of BRs on silica compound recipe with E-SBR and S-SBR and fine grade of silica.

Preparation and analysis have been done in the same way as in the section 5-8. All the data are summarized in Tables 40, 41, 42, 43 and 44 below.

Table 40

	C101 (Ref)	C102 (Ref)	C103 (Ref)	C104 (Ref)	C105 (Ref)	C106 (Ref)	C107 (Ref)	C108
E-SBR1502	70							
S-SBR E15		70	70	70	70	70	70	70
BR150L	30	30						
BR360L			30					
CB24				30				
NEOCIS-BR040					30			
LG-1208						30		
High ML CBR matrix (ML = 70)							30	
CBR215 (Coconut 21.5 phr)								36.45
Ultrasil7000GR	75	75	75	75	75	75	75	75
Si69	6	6	6	6	6	6	6	6
Sunthene oil 4240	21.5	21.5	21.5	21.5	21.5	21.5	21.5	15.05
ZnO	3	3	3	3	3	3	3	3
St Acid	1	1	1	1	1	1	1	1
AO.6C	1	1	1	1	1	1	1	1
	207.5	207.5	207.5	207.5	207.5	207.5	207.5	207.5
CBS	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
DPG	2	2	2	2	2	2	2	2
Sulfur	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4
Total	212.6	212.6	212.6	212.6	212.6	212.6	212.6	212.6

Table 41

	C101 (Ref)	C102 (Ref)	C103 (Ref)	C104 (Ref)	C105 (Ref)	C106 (Ref)	C107 (Ref)	C108
E-SBR1502	420							
S-SBR E15		420	420	420	420	420	420	420
BR150L	180	180						
BR360L			180					
CB24				180				
NEOCIS-BR040					180			
LG-1208						180		
High ML CBR matrix (ML = 70)							180	
CBR215 (Coconut 21.5 phr)								218.7
Ultrasil7000GR	450	450	450	450	450	450	450	450
Si69	36	36	36	36	36	36	36	36
Sunthene oil 4240	129	129	129	129	129	129	129	90.3
ZnO	18	18	18	18	18	18	18	18
St Acid	6	6	6	6	6	6	6	6
AO.6C	6	6	6	6	6	6	6	6
	1245	1245	1245	1245	1245	1245	1245	1245
	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5
CBS	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5
DPG	10	10	10	10	10	10	10	10
Sulfur	7	7	7	7	7	7	7	7
Total	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0	1063.0

Table 42

Grade name			C102 (Ref)	C103 (Ref)	C104 (Ref)	C105 (Ref)	C106 (Ref)	C107 (Ref)	C108
			BR150L	BR360L	CB24	BR040	LG1208	HM-BR	HM CBR21.5
Mooney viscosity		ML ₁₊₄ , 100	42.2	48.5	43.4	44.6	47.0	74.3	40.5
T-cp		(cps)	102	127.2	126.8	211.1	252.1	235.0	
T-cp/ML(1+4)			2.4	2.6	2.9	4.7	5.4	3.2	
Cis1,4			98.37	98.6	96.5	96.5	98.0	98.5	
Vinyl 1,2			0.8	0.8	0.4	0.4	1.1	0.8	
Tran 1,2			0.77	0.6	3.1	3.1	0.9	0.7	
MWD	Mw	(10 ⁻⁴)	52.99	57	46	54	58	74	
	Mn	(10 ⁻⁴)	21.9	23	21	22	16	24	
	Mw/Mn		2.42	2.45	2.20	2.40	3.60	3.01	

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Table 43

			E-SBR1502	150L	BR360L	CB24	BR040	LG1208	HM-BR	HM CBR21.5
Product name			C101 (Ref)	C102 (Ref)	C103 (Ref)	C104 (Ref)	C105 (Ref)	C106 (Ref)	C107 (Ref)	C108
Mooney viscosity; 1st		ML ₁₊₄ , 100	87.2	111	112	111.7	112.8	107.3	125.1	115.7
Mooney viscosity; 2nd		ML ₁₊₄ , 100°C	67.5	98.0	95.3	92.5	92.9	89.4	101.6	93.9
Curing rate	Min. T	(dN · m)	2.1	3.6	3.5	3.4	3.3	3.2	3.7	3.4
(150 °C)	Max. T	(dN · m)	19.9	20.3	21.1	20.4	20.4	20.2	21.4	21.8
	Ts1	min.	2.1	1.1	1.0	1.1	1.1	1.0	0.6	0.6
	Tc(10)	min.	3.1	1.3	1.3	1.4	1.4	1.3	1.3	1.4
	Tc(90)	min.	8.42	11.27	10.56	11.00	11.59	10.55	12.04	10.43

Table 44

			E-SBR1502	150L	BR360L	CB24	BR040	LG1208	HM-BR	HM CBR21.5
Product name			C101 (Ref)	C102 (Ref)	C103 (Ref)	C104 (Ref)	C105 (Ref)	C106 (Ref)	C107 (Ref)	C108
Hardness	Type A		66	68	69	69	69	69	71	70
Specific Gravity		-	1.187	1.189	1.189	1.190	1.190	1.188	1.189	1.190
100% Modulus		kg/cm ²	23	29	29	29	28	29	31	29
200% Modulus		kg/cm ²	54	72	72	73	68	72	78	72
300% Modulus		kg/cm ²	103	137	137	139	129	134	145	137
Tensile strength		kg/cm ²	149	169	151	158	152	151	158	165
Elongation		%	383	344	320	332	336	325	310	334
Tear resistance		kg/cm	65	59	58	59	65	60	64	62
Rebound	BS	%	48.2	50.4	55.2	55.2	56.2	53.6	56.2	58.5
vs Sunthene		Index	100	105	114	114	116	111	116	121
Akron Abrasion		cc loss	0.140	0.052	0.051	0.046	0.051	0.045	0.049	0.055
vs Sunthene		Index	37	100	102	113	102	116	106	95
Compression Set		%	21.8	14.4	14.2	14.1	14.8	16.8	15.3	14.6
vs Sunthene		Index	66	100	101	102	97	86	94	99
Cut Growth Resistance (ASTM D813 : 2-15 mm)	Stroke	Kcycle	35	36	97	123	72	61	87	233
57 mm		Index	97	100	269	343	199	169	241	648

As shown in Table 44, the physical properties are generally improved by using coconut oil extended BR compared to the case where other types of BRs are used.

5-12. The comparison of physical properties where coconut oil extended BRs (CBRs) with various ML viscosity level we employed

Preparation and analysis have been done in the same way as in the section 5-8. All the data are summarized in Tables 45, 46, 47, 48 and 49 below. Here, CBR50 means the CBR with ML viscosity of around 50 and the same rule applies to CBR60, CBR70, and CBR80. FFA contents for these are 21.5% by mass.

Table 45

	C109 (Ref)	C110 (Ref)	C111	C112	C113	C114
S-SBR E15	70	70	70	70	70	70
BR150L	30					
CB24		30				
CBR50			36.45			
CBR60				36.45		
CBR70					36.45	
CBR80						36.45
Ultrasil 7000GR	75	75	75	75	75	75
Si69	6	6	6	6	6	6
Sunthene oil 4240	21.5	21.5	15.05	15.05	15.05	15.05
ZnO	3	3	3	3	3	3
stearic acid	1	1	1	1	1	1
6PPD	1	1	1	1	1	1
	207.5	207.5	207.5	207.5	207.5	207.5
Compound	207.5	207.5	207.5	207.5	207.5	207.5
CBS	1.7	1.7	1.7	1.7	1.7	1.7
DPG	2.0	2.0	2.0	2.0	2.0	2.0
sulfur	1.4	1.4	1.4	1.4	1.4	1.4
	212.6	212.6	212.6	212.6	212.6	212.6

Table 46

	C109 (Ref)	C110 (Ref)	C111	C112	C113	C114
S-SBR E15	420	420	420	420	420	420
BR150L	180	0	0	0	0	0
CB24	0	180	0	0	0	0
CBR50	0	0	218.7	0	0	0
CBR60	0	0	0	218.7	0	0
CBR60	0	0	0	0	218.7	0
CBR70	0	0	0	0	0	218.7
Ultrasil 7000GR	450	450	450	450	450	450
Si69	36	36	36	36	36	36
Sunthene oil 4240	129	129	90.3	90.3	90.3	90.3
ZnO	18	18	18	18	18	18
stearic acid	6	6	6	6	6	6
6PPD	6	6	6	6	6	6
	1245	1245	1245	1245	1245	1245
Compound	1037.5	1037.5	1037.5	1037.5	1037.5	1037.5
CBS	8.5	8.5	8.5	8.5	8.5	8.5
GPG	10.0	10.0	10.0	10.0	10.0	10.0
sulfur	7.0	7.0	7.0	7.0	7.0	7.0
	1063	1063	1063	1063	1063	1063

Table 47

Grade name			C109 (Ref)	C110 (Ref)	C111	C112	C113	C114
			BR150L	CB24	CBR50	CBR60	CBR70	CBR80
Mooney viscosity		ML ₁₊₄ , 100	42.2	43.4	49.6	58.7	69.0	78.0
T-cp		(cps)	102.0	126.8	168.9	223.2	266.8	347.8
T-cp/ML(1+4)			2.4	2.9	3.4	3.8	3.9	4.5
Cis1,4			98.4	96.5	98.6	98.2	98.6	98.7
Vinyl 1,2			0.8	0.4	0.7	0.8	0.7	0.6
Tran 1,2			0.8	3.1	0.7	0.9	0.7	0.7
MWD	Mw	(10 ⁻⁴)	53	46	64	67	79	78
	Mn	(10 ⁻⁴)	22	21	30	32	35	36
	Mw/Mn		2.42	2.20	2.13	2.10	2.24	2.16

Table 48

Product name			C109 (Ref)	C110 (Ref)	C111	C112	C113	C114
Grade name			BR150L	CB24	CBR50	CBR60	CBR70	CBR80
Mooney viscosity; 1st		ML ₁₊₄ , 100	109.1	108.4	106.6	112.9	115.8	116.4
Mooney viscosity; 2nd		ML ₁₊₄ , 100°C	92.4	91.5	84.3	88.8	90.6	91.4
Curing rate	Min. T	(dN · m)	3.9	3.8	3.4	3.6	3.7	3.8
(150 °C)	Max. T	(dN · m)	22.2	22.3	23.5	23.4	24.3	24.4
	Ts1	min.	1.0	0.6	0.6	0.6	0.5	0.5
	Tc(10)	min.	1.3	1.3	1.5	1.5	1.4	1.4
	Tc(90)	min.	11.32	11.51	11.48	11.54	10.48	11.53

Table 49

Product name			C109 (Ref)	C110 (Ref)	C111	C112	C113	C114
Grade name			BR150L	CB24	CBR50	CBR60	CBR70	CBR80
Hardness	Type A		69	69	69	69	70	71
Specific Gravity		-	1.188	1.189	1.190	1.190	1.190	1.190
100% Modulus		kg/cm ²	30	30	29	30	30	30
200% Modulus		kg/cm ²	76	74	72	72	72	71
300% Modulus		kg/cm ²	142	139	133	136	134	134
Tensile strength		kg/cm ²	147	155	152	149	158	153
Elongation		%	308	322	327	318	337	330
Tear resistance		kg/cm	57	63	62	61	63	62
Rebound	BS	%	55.2	56.2	56.8	56.8	56.8	56.8
vs Sunthene		Index	100	102	103	103	103	103
Akron Abrasion		cc loss	0.059	0.061	0.066	0.061	0.060	0.062
vs Sunthene		Index	100	97	89	97	98	95
Compression Set		%	15.8	15.5	15.1	14.0	14.3	14.7
vs Sunthene		Index	100	102	105	113	110	107
Viscoelasticity								
-20°C								
E' (MPa)				60.2	59.9	65.2	65.7	64.6
E'' (MPa)				34.1	36.0	35.6	35.9	36.3
E* (MPa)				69.2	69.9	74.3	74.9	74.1
tand				0.566	0.601	0.545	0.546	0.562
Index (vs CB24)				100	106	96	97	99
0°C								
E' (MPa)				16.9	14.7	17.9	18.8	17.1
E'' (MPa)				4.3	3.6	4.1	4.3	4.0
E* (MPa)				17.4	15.1	18.3	19.3	17.6
tand				0.252	0.244	0.228	0.232	0.236
Index (vs Sunthene)				100	97	90	92	93
60°C								
E' (MPa)				8.5	8.3	9.7	10.1	9.2
E'' (MPa)				0.7	0.6	0.8	0.8	0.7
E* (MPa)				8.5	8.3	9.8	10.1	9.3
tand				0.081	0.077	0.079	0.080	0.080
Index (vs Sunthene)				100	105	103	101	101

As shown in Table 49, the physical properties are generally improved by using coconut oil extended BR regardless of their Mooney viscosities.

5-13. The effect of coconut oil extended BR on Truck Bus tread formulation with carbon black compound

5-13-1. Non-Productive Mixing (Primary Compound)

During the non-productive mixing, all components except the vulcanizing agent and accelerators were mixed in the standard mixer such as a banbury mixer with initial temperature at 90 °C within 5 minutes mixing time. Firstly, all of diene polymers were added into banbury mixer and mixed for 1 minute, then, all of filler was added into mixer. All of mixtures were mixed in banbury mixer for 2.5 minutes. Then, ram of mixer chamber was opened up for cleaning residue filler at 2.5 minutes of mixing process. The mixing process had proceeded for 5 minutes or temperature reached 170 °C. The mixed compounds were rolled at preferred temperature range of 60-70 °C using the standard roller with nip clearance of 2 millimeter. The samples of the compound sheets were left at room temperature for 1-24 hours. Then, the compound samples were subject to the Mooney viscosity measurement.

The compositions are summarized in Tables 50 and 51 below, the former of which is described in phr and the latter of which is described in grams. The properties of various BRs are summarized in Table 52 below.

Table 50

	C 115 (Ref)	C 116 (Ref)	C 117 (Ref)	C 118 (Ref)	C 119 (Ref)	C 120 (Ref)	C 121 (Ref)	C 122
CB24	25							
NEODENE-45		25						
NeocisBR040			25					
LG-1208				25				
BR150L					25			
BR360L						25		
BR230							25	
CBR21.5								25
NR STR20 (ML = 70)	75	75	75	75	75	75	75	75
SAF-C/B (N234)	50	50	50	50	50	50	50	50
VIVATEC500	3	3	3	3	3	3	3	3
ZnO	3	3	3	3	3	3	3	3
stearic acid	2	2	2	2	2	2	2	2
6PPD	2	2	2	2	2	2	2	2
	160	160	160	160	160	160	160	160
Compound	160	160	160	160	160	160	160	160
NS	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
sulfur	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
	162.5	162.5	162.5	162.5	162.5	162.5	162.5	162.5

Table 51

	C 115 (Ref)	C 116 (Ref)	C 117 (Ref)	C 118 (Ref)	C 119 (Ref)	C 120 (Ref)	C 121 (Ref)	C 122
CB24	187.5	0	0	0	0	0	0	0
NEODENE-45	0	187.5	0	0	0	0	0	0
NeocisBR040	0	0	187.5	0	0	0	0	0
LG-1208	0	0	0	187.5	0	0	0	0
BR150L	0	0	0	0	187.5	0	0	0
BR360L	0	0	0	0	0	187.5	0	0
BR230	0	0	0	0	0	0	187.5	0
CBR21.5	0	0	0	0	0	0	0	187.5
NR (ML = 70)	562.5	562.5	562.5	562.5	562.5	562.5	562.5	562.5
SAF-C/B (N234)	375	375	375	375	375	375	375	375
VIVATEC500	22.5	22.5	22.5	22.5	22.5	22.5	22.5	22.5
ZnO	22.5	22.5	22.5	22.5	22.5	22.5	22.5	22.5
stearic acid	15	15	15	15	15	15	15	15
6PPD	15	15	15	15	15	15	15	15
	1200	1200	1200	1200	1200	1200	1200	1200
Compound	800.0	800.0	800.0	800.0	800.0	800.0	800.0	800.0
NS (TBBS)	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
sulfur	7.50	7.50	7.50	7.50	7.50	7.50	7.50	7.50
	812.5	812.5	812.5	812.5	812.5	812.5	812.5	812.5

Table 52

Grade name			CB24	NEODENE-45	BR040	LG-1208	BR150L	BR360L	BR230	CBR21.5
Mooney viscosity		ML ₁₊₄ , 100	43.4	44.0	44.6	47.0	42.2	48.5	34.5	39.0
T-cp		(cps)	126.8	216.3	211.1	252.1	102	127.2	118.7	N/A
T-cp/ML(1+4)			2.9	4.9	4.7	5.4	2.4	2.6	3.4	N/A
Cis1,4			96.5	97.6	96.5	98.0	98.4	98.6	98.3	98.6*
Vinyl 1,2			0.4	0.5	0.4	1.1	0.8	0.8	0.8	0.8*
Tran 1,2			3.1	1.9	3.1	0.9	0.8	0.6	0.9	0.6*
MWD	Mw	(10 ⁻⁴)	46.2	58.9	54.0	58.3	52.99	57.1	56.7	73.5*
	Mn	(10 ⁻⁴)	21.0	21.2	22.2	16.0	21.90	23.3	14.7	24.4*
	Mw/Mn		2.2	2.8	2.4	3.6	2.42	2.4	3.9	3.0*

* values are based on rubber portion only.

5-13-2. Productive Mixing (Secondary Compound)

- 5 The sheets of the primary compounds obtained from the aforementioned non-productive mixing were then subject to the mixing with vulcanizing agent, most preferably sulfur, and the vulcanizing accelerators by using the standard roll at preferred temperature range of 60-70 °C within 3 minutes. The rubber compounds from the productive mixing (secondary compound) have been pulled in sheets and the samples were then subject to the measurements of Mooney
- 10 viscosity (ML1+4,100 °C), curing time on a Moving Die Rheometer (MDR) at 160 °C.

The results are summarized in Table 53 below.

Table 53

Product name			C 115 (Ref)	C 116 (Ref)	C 117 (Ref)	C 118 (Ref)	C 119 (Ref)	C 120 (Ref)	C 121 (Ref)	C 122
Grade name			CB24	NEODENE-45	NeocisBR040	LG-1208	BR150L	BR360L	BR230	CBR21.5
Mooney viscosity; 2nd		ML1+4,100°C	79.8	80.7	77.5	79.3	77.1	81.8	73.0	71.7
Curing rate	Min. T	(dN · m)	3.2	3.3	3.1	3.2	3.2	3.2	3.0	3.1
(150 °C)	Max. T	(dN · m)	19.6	19.4	19.7	19.1	19.4	19.4	18.5	18.3
	Ts1	min.	2.4	2.4	2.4	2.4	2.4	2.3	2.4	2.4
	Tc(10)	min.	3.1	3.1	3.1	3.1	3.1	3.0	3.1	3.1
	Tc(90)	min.	7.43	7.53	7.37	7.40	7.38	7.29	7.33	7.36

5-13-3. Vulcanization and Properties of the Filler-Filled Vulcanizates

The secondary filler-filled rubber compounds obtained from the productive mixing were processed in the mold pressing at 150 °C according the curing time observed by a MDR as already mentioned (t90x2). The rubber vulcanizates in the present invention in various forms of the specimens were then subject to the measurements of the viscoelastic property during the temperature sweep, tensile strength, hardness, specific gravity, tear resistance, rebound resilience, abrasion resistance, and compression set.

The results are summarized in Table 54 below.

Table 54

Product name			C 115 (Ref)	C 116 (Ref)	C 117 (Ref)	C 118 (Ref)	C 119 (Ref)	C 120 (Ref)	C 121 (Ref)	C 122
Grade name			CB24	NEODENE-45	NeocisBR040	LG-1208	BR150L	BR360L	BR230	CBR21.5
Hardness	Type A		66-67	67	67	66-67	67	67	66	65
Specific Gravity		-	1.108	1.108	1.108	1.109	1.108	1.108	1.108	1.109
100% Modulus		kg/cm ²	29	28	29	28	30	30	28	28
200% Modulus		kg/cm ²	76	73	74	71	77	78	73	72
300% Modulus		kg/cm ²	138	137	137	132	141	142	136	133
Tensile strength		kg/cm ²	300	283	293	295	310	299	298	276
Elongation		%	545	512	533	547	554	536	553	527
(TBxEB)/2			81,750	72,448	78,041	80,683	85,870	80,132	82,397	72,726
Tear resistance		kg/cm	87	82	81	85	84	83	83	82
Rebound	BS	%	58.5	58.5	58.5	56.8	58.5	60.1	56.8	60.1
vs BR150L		Index	100	100	100	97	100	103	97	103
Akron Abrasion		cc loss	0.044	0.043	0.053	0.043	0.044	0.041	0.045	0.053
vs BR150L		Index	100	102	83	102	100	107	98	83
Compression Set		%	29.0	27.2	29.9	28.6	27.7	25.6	27.9	25.8
vs BR150L		Index	100	107	97	101	105	113	104	112
Heat Build Up										
D T		(°C)	22.0	22.2	22.0	23.0	22.2	21.7	23.3	20.8
vs BR150L		Index	101	100	101	97	100	102	95	107
PS		(%)	10.0	9.3	11.2	10.6	10.1	9.1	12.0	9.8
vs BR150L		Index	101	109	90	95	100	111	84	103
Viscoelasticity										
-20°C										
E' (MPa)			24.3	22.3	24.7	22.6	24.8	24.0	22.1	25.5
E'' (MPa)			5.9	5.4	6.0	5.6	6.0	6.0	5.5	6.4
E* (MPa)			25.0	23.0	25.5	23.3	25.5	24.8	22.8	26.3
tand			0.244	0.243	0.242	0.246	0.243	0.252	0.249	0.253
Index (vs BR150L)			100	100	99	101	100	103	102	104
0°C										
E' (MPa)			17.6	16.4	18.2	16.5	18.0	17.3	16.1	17.1
E'' (MPa)			2.6	2.4	2.7	2.5	2.7	2.6	2.5	2.7
E* (MPa)			17.81	16.56	18.37	16.67	18.16	17.54	16.27	17.27
tand			0.148	0.146	0.149	0.151	0.151	0.151	0.152	0.160
Index (vs BR150L)			98	97	99	100	100	100	101	106
60°C										
E' (MPa)			11.6	10.9	11.8	10.7	11.7	11.3	10.5	11.0
E'' (MPa)			1.2	1.1	1.2	1.1	1.2	1.2	1.1	1.1
E* (MPa)			11.6	11.0	11.8	10.8	11.8	11.4	10.6	11.0
tand			0.101	0.097	0.103	0.102	0.103	0.102	0.104	0.102
Index (vs BR150L)			103	107	101	102	100	102	99	101

As shown in Table 54, the physical properties are generally improved by using coconut oil extended BR compared to the case where other types of BRs are employed.

5-13-4. Processability

The processability is also studied by applying several rates of shears at the temperature of 120 °C. The results are summarized in Figures 2 and 3.

As is clear from Figures 2 and 3, the processability is also improved by using coconut oil extended BR.

- 5 5-14. The effect of coconut oil extended BR on Truck Bus tread hybrid formulation with silica and carbon black compound

5-14-1. Non-Productive Mixing (Primary Compound)

During the non-productive mixing, all components except the vulcanizing agent and accelerators were mixed in the standard mixer such as a banbury mixer with initial temperature at 90 °C within 5 minutes mixing time. Firstly, all of mixtures of diene polymers were mixed in banbury mixer for 30 seconds. Then, half of filler especially silica and silane coupling agent were added in to mixer. At 1 minute and 30 seconds of mixing process, all of fillers and other rubber compound ingredients were added into mixer. Then, ram of mixer chamber was opened up for cleaning residue filler. The mixing process had proceeded for 5 minutes. When mixing temperature reached 145 °C, the rotor speed of mixer had been reduced. The mixed compounds were rolled at preferred temperature range of 60-70 °C using the standard roller with nip clearance of 2 millimeter. The samples of the compound sheets were subject to the Mooney viscosity measurement.

The compositions are summarized in Tables 55 and 56 below, the former of which is described in phr and the latter of which is described in grams. The properties of various BRs are summarized in Table 52 above.

Table 55

	C 123(Ref)	C 124 (Ref)	C 125 (Ref)	C 126 (Ref)	C 127 (Ref)	C 128 (Ref)	C 129 (Ref)	C 130	C 131 (Ref)	C 132 (Ref)
CB24	25									
NEODENE-45		25								
NeocisBR040			25							
LG-1208				25						
BR150L					25				25	40
BR360L						25				
BR230							25			
CBR21.5								30.375		
NR STR20 (ML = 70)	75	75	75	75	75	75	75	75	75	60
SAF-C/B (N234)	30	30	30	30	30	30	30	30	10	30
Ultrasil 7000GR	20	20	20	20	20	20	20	20	40	20
Si69	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	3.2	1.6
VIVATEC500	3	3	3	3	3	3	3	0	3	3
ZnO	3	3	3	3	3	3	3	3	3	3
stearic acid	2	2	2	2	2	2	2	2	2	2
6PPD	2	2	2	2	2	2	2	2	2	2
	161.6	161.6	161.6	161.6	161.6	161.6	161.6	163.975	163.2	161.6
Compound	161.6	161.6	161.6	161.6	161.6	161.6	161.6	163.975	163.2	161.6
NS	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
sulfur	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
	164.1	164.1	164.1	164.1	164.1	164.1	164.1	166.475	165.7	164.1

Table 56

	C 123(Ref)	C 124 (Ref)	C 125 (Ref)	C 126 (Ref)	C 127 (Ref)	C 128 (Ref)	C 129 (Ref)	C 130	C 131 (Ref)	C 132 (Ref)
CB24	187.5	0	0	0	0	0	0	0	0	0
NEODENE-45	0	187.5	0	0	0	0	0	0	0	0
NeocisBR040	0	0	187.5	0	0	0	0	0	0	0
LG-1208	0	0	0	187.5	0	0	0	0	0	0
BR150L	0	0	0	0	187.5	0	0	0	187.5	300
BR360L	0	0	0	0	0	187.5	0	0	0	0
BR230	0	0	0	0	0	0	187.5	0	0	0
CBR21.5	0	0	0	0	0	0	0	227.813	0	0
NR STR20 (ML = 70)	562.5	562.5	562.5	562.5	562.5	562.5	562.5	562.5	562.5	450
SAF-C/B (N234)	225	225	225	225	225	225	225	225	75	225
Ultrasil 7000GR	150	150	150	150	150	150	150	150	300	150
Si69	12	12	12	12	12	12	12	12	24	12
VIVATEC500	22.5	22.5	22.5	22.5	22.5	22.5	22.5	0	22.5	22.5
ZnO	22.5	22.5	22.5	22.5	22.5	22.5	22.5	22.5	22.5	22.5
stearic acid	15	15	15	15	15	15	15	15	15	15
6PPD	15	15	15	15	15	15	15	15	15	15
	1212	1212	1212	1212	1212	1212	1212	1229.81	1224	1212
Compound	1050.4	1050.4	1050.4	1050.4	1050.4	1050.4	1050.4	1065.8	1060.8	1050.4
NS (TBBS)	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5
sulfur	9.8	9.8	9.8	9.8	9.8	9.8	9.8	9.8	9.8	9.8
	1066.65	1066.7	1066.65	1066.65	1066.65	1066.65	1066.65	1082.09	1077.1	1066.7

5-14-2. Productive Mixing (Secondary Compound)

- 5 The sheets of the primary compounds obtained from the aforementioned non-productive mixing were then subject to the mixing with vulcanizing agent, most preferably sulfur, and the vulcanizing accelerators by using the standard roll at preferred temperature range of 60-70 °C within 4 minutes. The rubber compounds from the productive mixing (secondary compound) have been pulled in sheets and the samples were then subject to the measurements of Mooney
- 10 viscosity (ML1+4,100 °C), curing time on a Moving Die Rheometer (MDR) at 160 °C.

The results are summarized in Table 57 below.

Table 57

Product name			C 123(Ref)	C 124 (Ref)	C 125 (Ref)	C 126 (Ref)	C 127 (Ref)	C 128 (Ref)	C 129 (Ref)	C 130	C 131 (Ref)	C 132 (Ref)
Grade name			CB24	NEODENE-45	NeocisBR040	LG-1208	BR150L	BR360L	BR230	CBR21.5	150L/25	150L/40
Mooney viscosity; 2nd		ML1+4,100°C	69.0	69.6	69.1	67.6	67.8	69.7	63.7	65.0	66.0	73.7
Curing rate	Min. T	(dN · m)	2.6	2.6	2.6	2.7	2.7	2.8	2.6	2.5	2.6	2.9
(150 °C)	Max. T	(dN · m)	15.7	15.6	16.0	16.17	16.0	16.4	15.3	15.1	13.7	17.0
	Ts1	min.	2.5	2.5	2.5	2.4	2.4	2.4	2.4	2.5	3.4	2.5
	Tc(10)	min.	3.1	3.1	3.1	2.6	3.0	3.0	3.0	3.1	3.5	3.1
	Tc(90)	min.	9.28	9.28	9.40	9.22	9.18	9.17	9.30	9.48	18.01	10.15

5-14-3. Vulcanization and Properties of the Filler-Filled Vulcanizates

The secondary filler-filled rubber compounds obtained from the productive mixing were processed in the mold pressing at 150 °C according the curing time observed by a MDR as already mentioned (t90x2). The rubber vulcanizates in the present invention in various forms of the specimens were then subject to the measurements of the viscoelastic property during the temperature sweep, tensile strength, hardness, specific gravity, tear resistance, rebound resilience, abrasion resistance, and compression set.

The results are summarized in Table 58 below.

10 Table 58

Product name			C 123(Ref)	C 124 (Ref)	C 125 (Ref)	C 126 (Ref)	C 127 (Ref)	C 128 (Ref)	C 129 (Ref)	C 130	C 131 (Ref)	C 132 (Ref)
Grade name			CB24	NEODENE-45	NeocisBR040	LG-1208	BR150L	BR360L	BR230	CBR21.5	150L/25	150L/40
Hardness	Type A		64	64	65	64	64	64	65	64	61	66
Specific Gravity		-	1.119	1.121	1.120	1.120	1.121	1.121	1.119	1.114	1.130	1.120
100% Modulus		kg/cm ²	21	21	21	22	21	21	21	21	15	21
200% Modulus		kg/cm ²	50	51	51	51	50	51	50	49	33	48
300% Modulus		kg/cm ²	95	97	96	95	94	96	93	93	61	90
Tensile strength		kg/cm ²	284	280	292	273	277	269	279	272	232	271
Elongation		%	624	623	639	615	634	591	628	615	673	631
Breaking Energy		(TBxEB)/2	88,608	87,220	93,294	83,948	87,809	79,490	87,606	83,640	78,068	85,501
Tear resistance		kg/cm	73	68	69	71	67	68	69	66	59	71
Rebound	BS	%	58.5	58.5	56.8	56.8	57.8	58.5	55.2	60.1	56.8	58.5
vs BR150L		Index	101	101	98	98	100	101	95	104	98	101
Akron Abrasion		cc loss	0.075	0.073	0.074	0.073	0.075	0.074	0.080	0.070	0.123	0.069
vs BR150L		Index	100	103	101	103	100	101	94	107	61	109
Lambourn abrasion												
20% slip rate		cc loss (g)	0.1004	0.0966	0.0920	0.0956	0.0934	0.0920	0.0958	0.0747	0.0853	0.0805
vs BR150L		Index	93	97	102	98	100	94	97	125	110	116
40% slip rate		cc loss (g)	0.0887	0.0880	0.0895	0.0933	0.0922	0.0876	0.0905	0.0766	0.0920	0.0621
vs BR150L		Index	104	105	103	99	100	105	102	120	100	149
Compression Set		%	22.6	22.9	22.7	23.4	23.4	24.7	25.7	24.5	26.5	23.6
vs BR150L		Index	103.5	102.2	103.1	100.0	100.0	94.7	91.1	95.5	88.3	99.2
Heat Build Up												
D T		(°C)	25.0	25.8	25.4	29.8	26.3	26.5	27.6	22.8	43.3	29.8
vs BR150L		Index	105	102	104	88	100	99	95	115	61	88
PS		(%)	13.0	12.3	12.0	14.3	12.6	12.5	14.3	10.5	34.8	13.6
vs BR150L		Index	97	103	105	88	100	101	88	120	36	93
Viscoelasticity												
-20°C												
E' (MPa)			15.6	15.4	15.3	16.4	16.8	16.0	16.7	17.5	12.5	14.6
E'' (MPa)			4.2	4.0	4.0	4.3	4.4	4.2	4.3	4.3	3.3	3.5
E* (MPa)			16.1	15.9	15.8	16.9	17.3	16.5	17.2	18.0	13.0	15.0
tand			0.270	0.260	0.261	0.261	0.263	0.261	0.257	0.249	0.265	0.237
Index (vs BR150L)			103	99	99	99	100	99	98	94	101	90
0°C												
E' (MPa)			11.1	11.0	11.0	11.6	12.0	11.4	11.9	11.3	8.7	10.6
E'' (MPa)			1.7	1.6	1.7	1.8	1.8	1.7	1.8	1.7	1.3	1.6
E* (MPa)			11.18	11.14	11.12	11.78	12.12	11.52	11.99	11.39	8.79	10.75
tand			0.151	0.149	0.151	0.152	0.152	0.150	0.155	0.152	0.147	0.148
Index (vs BR150L)			99	98	99	100	100	99	102	100	97	97
60°C												
E' (MPa)			7.2	7.2	7.1	7.7	7.6	7.4	7.7	7.1	5.7	7.5
E'' (MPa)			0.7	0.7	0.7	0.8	0.8	0.7	0.8	0.7	0.6	0.8
E* (MPa)			7.2	7.2	7.2	7.8	7.7	7.4	7.7	7.1	5.8	7.5
tand			0.100	0.099	0.100	0.101	0.102	0.099	0.106	0.095	0.111	0.104
Index (vs BR150L)			101	103	102	101	100	103	96	108	92	98

As shown in Table 58, the physical properties are generally improved by using coconut oil extended BR compared to the case where other types of BRs are employed.

5-14-4. Processability

The processability is also studied by the same way as described in section 5-13-4 and the results are summarized in Figures 4 and 5.

As is clear from Figures 4 and 5, the processability is also improved by using coconut oil
5 extended BR.

Best Mode for Carrying out the Invention

As we mentioned on the Invention disclosure section.

Industrial Applicability

The purpose of this invention is to provide an oil-extended rubber which has improved
10 physical properties and a rubber composition containing the oil-extended rubber, which can be
applied to rubber industry or tires industry or shoe sole industry containing the rubber
composition.

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Claims

1. An oil-extended rubber comprising:
 - a vulcanizable rubber component;
 - a coconut oil with a free fatty acid content of 0.05% by mass or more; and
- 5 wherein the content of the coconut oil is ranging from 21.5 to 37.5 part per hundred rubber (phr).
2. The oil-extended rubber according to claim 1, wherein an iodine value of the coconut oil is 10 or more.
- 10 3. The oil-extended rubber according to claim 1, wherein the free fatty acid content falls in a range of 2 to 18% by mass.
4. The oil-extended rubber according to claim 1, wherein the vulcanizable rubber component is a
- 15 polybutadiene.
5. The oil-extended rubber according to claim 1, wherein the vulcanizable rubber component is a 1,4-cis-polybutadiene.
- 20 6. A rubber composition comprising the oil-extended rubber according to any of the claims 1 to 5, and further comprising:
 - a diene-based rubber other than the vulcanizable rubber; and
 - a rubber reinforcing agent.
- 25 7. The rubber composition according to claim 6, further comprising a rubber process oil.
8. The rubber composition according to claim 6, further comprising a coconut oil.
9. The rubber composition according to any of claims 6 to 8, wherein the rubber reinforcing
- 30 agent comprises silica.
10. A rubber composition comprising a vulcanizable rubber component, a coconut oil with a free fatty acid content of 0.05% by mass or more, a diene-based rubber other than the vulcanizable

rubber, and a rubber reinforcing agent, wherein the content of the coconut oil is ranging from 21.5 to 37.5 part per hundred rubber (phr).

11. A tire comprising the rubber composition according to any one of the claims 6 to 10.

5

12. A shoe sole comprising the rubber composition according to any one of the claims 6 to 10.

13. A method for manufacturing an oil-extended rubber, the method comprising a step of:

10 mixing a vulcanizable rubber component and a coconut oil with a free fatty acid content
 of 0.05% by mass or more.

14. The method according to claim 13, wherein the mixing step is performed without adding solvents.

15 15. The method according to claim 13, further comprising a step of:

 dissolving the vulcanizable rubber component in a solvent prior to performing the mixing
 step; and
 using the dissolved vulcanizable rubber component in the mixing step.

20

Abstract

The purpose of this invention is to provide an oil-extended rubber which has improved physical properties and a rubber composition containing the oil-extended rubber as a component. An oil-extended rubber containing a vulcanizable rubber component and a coconut oil with a free fatty acid content of 0.05% by mass or more, particularly 5-15% by mass, has been provided for this purpose.

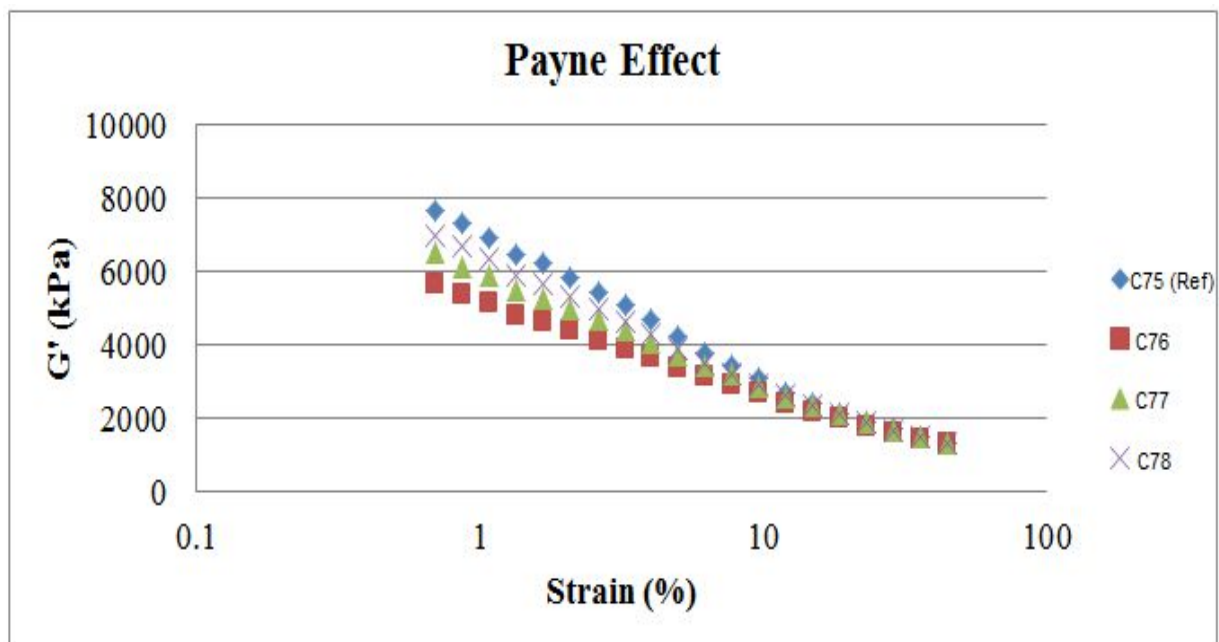
Drawings

Fig. 1

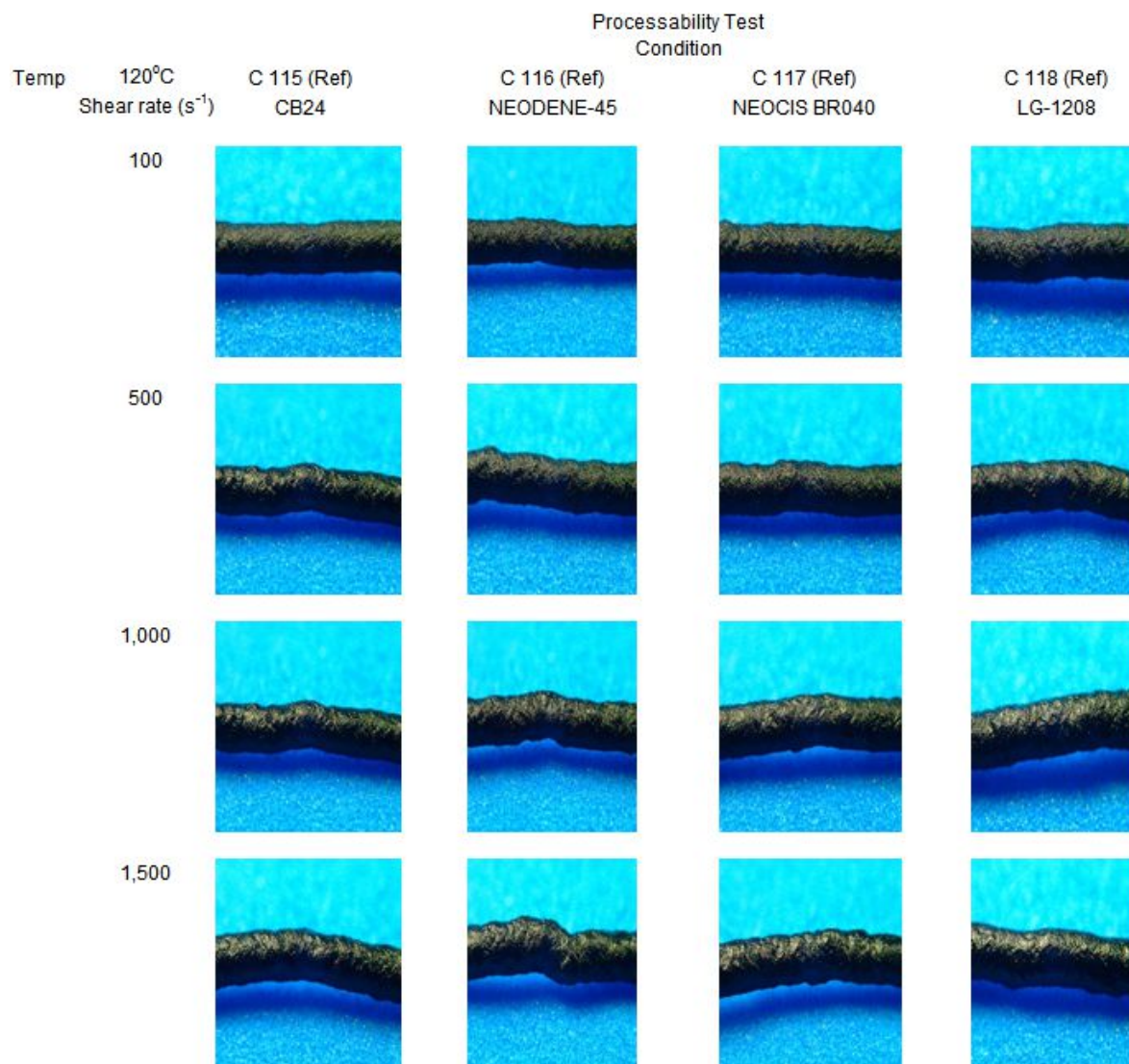


Fig. 2

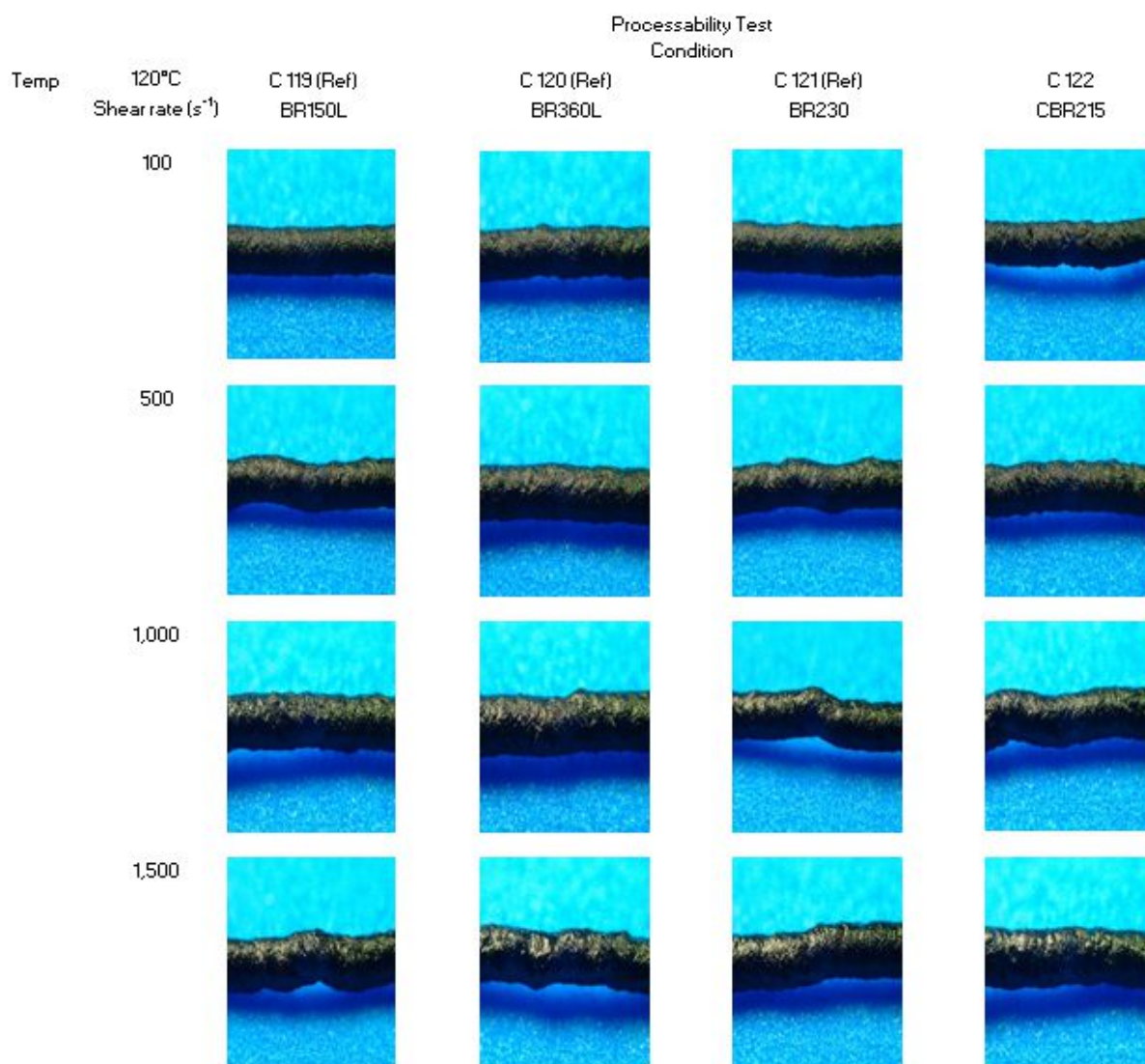


Fig. 3

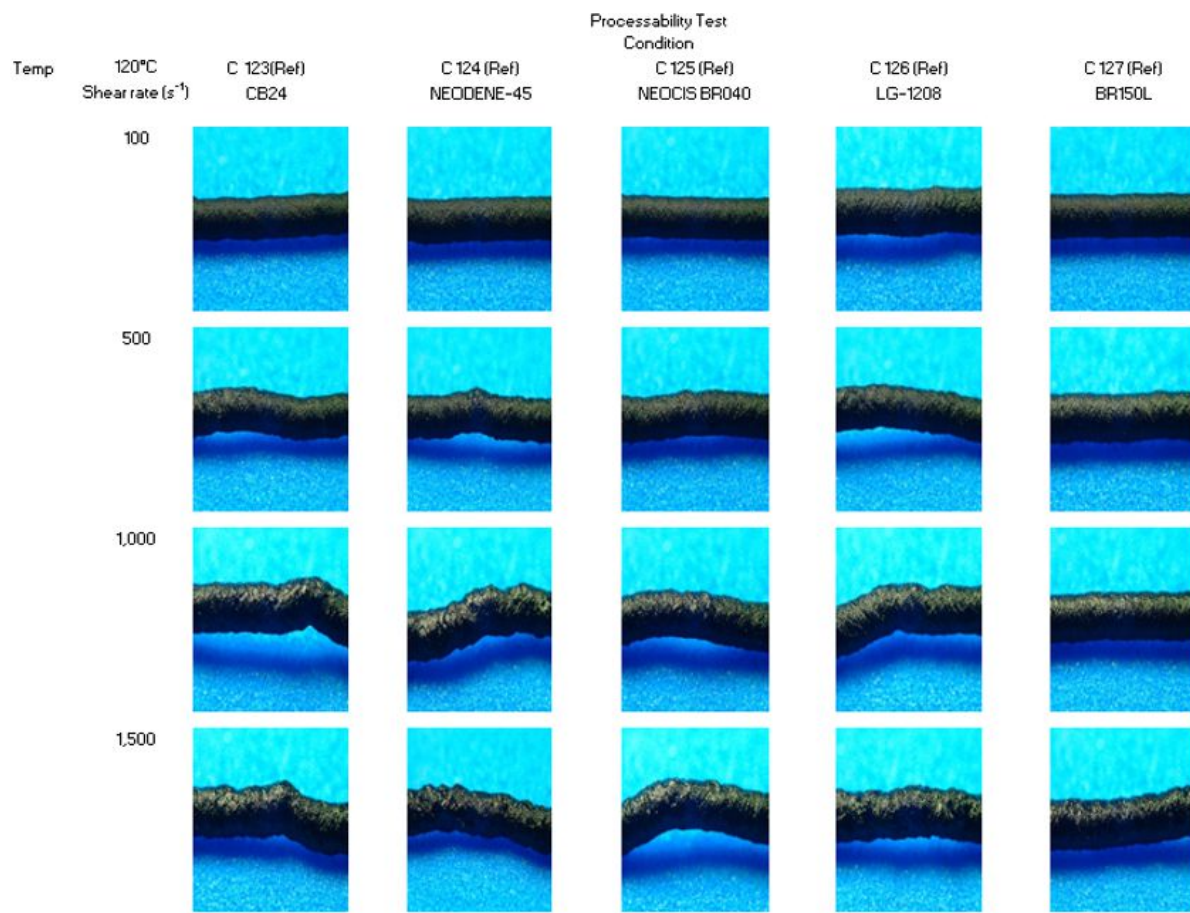


Fig. 4

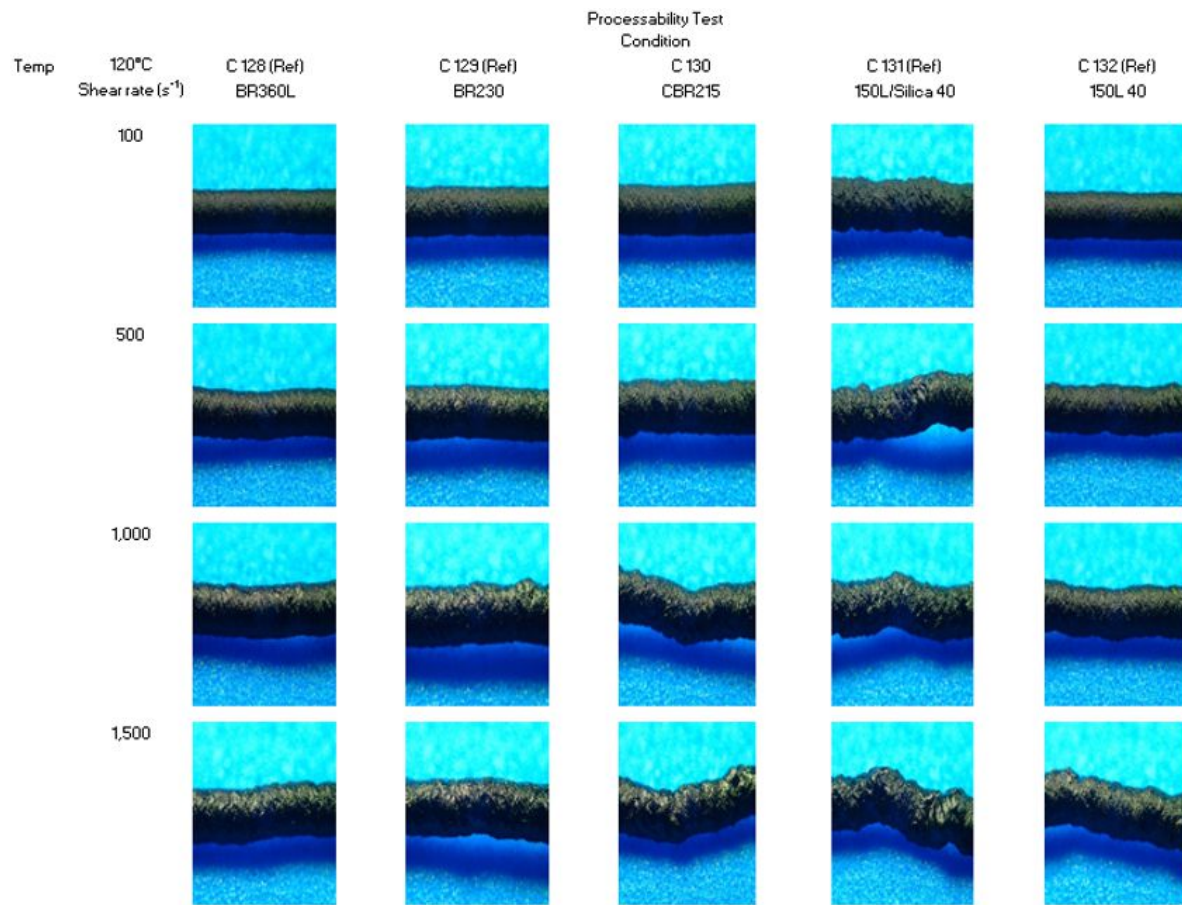


Fig. 5