



(51) International Patent Classification:

C08J 3/00 (2006.01)

C08L 7/00 (2006.01)

C08J 7/02 (2006.01)

C08L 21/00 (2006.01)

Published:

- with international search report (Art. 21(3))
- before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))

(21) International Application Number:

PCT/US2024/062069

(22) International Filing Date:

27 December 2024 (27.12.2024)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

63/615,437

28 December 2023 (28.12.2023) US

(71) Applicant: BRIDGESTONE CORPORATION [JP/JP];

1-1, Kyobashi 3-chome, Chuo-ku, Tokyo, 104-8340 (JP).

(72) Inventors; and

(71) Applicants: KIM, Hyeonjae [—/US]; 37 Garnett Circle,

Copley, OH 443 (US). HARTZELL, Michael [—/US];

4404 S. Primrose Drive, Gold Canyon, AZ 85118 (US).

(74) Agent: CHRISMAN, J., Gregory et al.; Chrisman Gal-

lo Tochtrop Llc, 6509 Brecksville Road, #31550, Indepen-

dence, OH 44131-4854 (US).

(81) Designated States (unless otherwise indicated, for every

kind of national protection available): AE, AG, AL, AM,

AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ,

CA, CH, CL, CN, CO, CR, CU, CV, CZ, DE, DJ, DK, DM,

DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT,

HN, HR, HU, ID, IL, IN, IQ, IR, IS, IT, JM, JO, JP, KE, KG,

KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY,

MA, MD, MG, MK, MN, MU, MW, MX, MY, MZ, NA,

NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO,

RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH,

TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS,

ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every

kind of regional protection available): ARIPO (BW, CV,

GH, GM, KE, LR, LS, MW, MZ, NA, RW, SC, SD, SL, ST,

SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ,

RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ,

DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT,

LU, LV, MC, ME, MK, MT, NL, NO, PL, PT, RO, RS, SE,

SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN,

GQ, GW, KM, ML, MR, NE, SN, TD, TG).

(54) Title: RUBBER BLENDS CONTAINING REGENERATED RUBBER

(57) Abstract: A method of preparing a rubber blend product that includes mixing regenerated rubber in a solvent to produce swollen regenerated rubber that is further blended with a mixture of another non-regenerated rubber such as natural rubber and an additional solvent to form a rubber blend mixture. The rubber blend mixture includes portions of the swollen regenerated rubber intertwined with the non-regenerated rubber. The rubber blend mixture is processed to remove portions of the solvents to form a rubber blend product for incorporation into rubber compositions for use in tires.



RUBBER BLENDS CONTAINING REGENERATED RUBBERTECHNICAL FIELD

[001] The present disclosure relates to rubber blends prepared by mixing a regenerated rubber and another rubber compound, such as a natural rubber, and more particularly, the present disclosure relates to swelling a regenerated rubbers in a solvent prior to combining with a non-regenerated rubber to form a rubber blend that includes a matrix of regenerated rubber and non-regenerated rubber.

BACKGROUND

[002] Commercial tires contain high amounts of materials that are derived from petroleum resources. Petroleum resources are limited in supply and therefore do not offer sustainable, predictable sources that can reduce carbon footprint. The present invention aims to significantly reduce reliance on non-sustainable resources and provide a blended rubber product for use in compositions for a tire, in which the use of petroleum resources and non-recycled materials are reduced for improving environmental friendliness of tires.

[003] There are many examples of recycled materials that can be used to increase material circularity in tire production. One of them is regenerated rubber from end-of-life tires. Depending on the process for manufacturing the regenerated rubber, it can be classified as one of the following three: reclaim rubber, ground rubber, or devulcanized rubber. However, these regenerated rubber products tend to lower physical properties of a rubber composition compound when added. In particular, weak interfaces between the matrix rubber and the regenerated rubber is a main contributor to reduction of tensile properties at high temperatures.

[004] The present invention focuses on a process for improving interactions between the matrix rubber and the regenerated rubber of either cured particles, crumbs and/or molecules or partially devulcanized rubber by using a solution mixing technology.

SUMMARY

[005] In a first aspect, disclosed is a method for preparing a rubber blend product, the method includes mixing a regenerated rubber and a first solvent to form a regenerated rubber slurry; separately mixing a rubber component, such as a synthetic rubber, natural rubber and/or a non-regenerated rubber, and a second solvent to form a rubber mixture; blending the regenerated rubber slurry and the rubber mixture to form a rubber blend mixture; and further removing a portion of the first solvent and a portion of the second solvent from the rubber blend mixture to form a rubber blend product.

[006] In an example of aspect 1, the regenerated rubber and/or the rubber (e.g., a non-regenerated rubber) are solid when mixed with the first solvent or second solvent.

[007] In another example of aspect 1, the regenerated rubber slurry includes the regenerated rubber in a solids content in the range of 1-15 weight percent based on the total weight of the regenerated rubber slurry.

[008] In another example of aspect 1, the blending of the regenerated rubber slurry and the rubber mixture is carried out under low shear conditions.

[009] In another example of aspect 1, the blending of the regenerated rubber slurry and the rubber mixture is carried out in a water bath for controlling the temperature of the rubber blend mixture. In other examples, the temperature of the rubber blend can be controlled in method known in the art, for instance, steam jacketed vessel, water heated jacketed vessel, and the like.

[0010] In another example of aspect 1, the blending of the regenerated rubber slurry and the rubber mixture, arranged in a container, is carried out by shaking, agitating or rotating the container.

[0011] In another example of aspect 1, the regenerated rubber slurry contains only the regenerated rubber and the first solvent. In an example, the regenerated rubber slurry is free of water and is a non-aqueous slurry.

[0012] In another example of aspect 1, the regenerated rubber slurry is formed by mixing the components at a temperature in the range of 10-55 °C or 15-35 °C and/or at a pressure in the range of 2.75 to 115 psia.

[0013] In another example of aspect 1, the regenerated rubber in the regenerated rubber slurry swells in the first solvent to form swollen regenerated rubber (e.g., particles or molecules). The swollen regenerated rubber provides open areas in the swollen matrix for incorporating portions of the rubber in the rubber mixture.

[0014] In another example of aspect 1, the swollen regenerated rubber occupies more volume as compared to non-swollen and/or the starting dry regenerated rubber used during mixing with the first solvent for forming the regenerated rubber slurry.

[0015] In another example of aspect 1, the rubber of the rubber mixture penetrates or becomes entangled with one or more portions of the swollen regenerated rubber of the regenerated rubber slurry during blending. The blending forms intertwined material that includes a swollen regenerated rubber intertwined with a rubber.

[0016] In another example of aspect 1, the rubber is a natural rubber, a natural rubber derived from a guayule plant or is a guayule natural rubber cement. In an example, the guayule natural rubber cement is fed from separate vessel to a tank for forming the rubber mixture.

[0017] In another example of aspect 1, the rubber mixture includes the rubber in a solids content in the range of 5-30 or 5-15 weight percent based on the total weight of the rubber mixture.

[0018] In another example of aspect 1, the rubber mixture contains only natural rubber and the first solvent or only a non-regenerated rubber and the first solvent.

[0019] In another example of aspect 1, the rubber mixture is formed at a temperature in the range of 15-55 °C and/or at a pressure in the range of 2.75 to 115 psia.

[0020] In another example of aspect 1, the first solvent and/or the second solvent includes a hydrocarbon solvent. In an example, the hydrocarbon solvent is hexane or butene. In an example, the first and/or second solvent is an organic solvent or a non-aqueous solvent.

[0021] In another example of aspect 1, the second solvent includes a ketone solvent or a polar solvent. In an example, the ketone or polar solvent is acetone.

[0022] In another example of aspect 1, the rubber blend mixture includes a weight ratio of the rubber to the regenerated rubber of 2:1 to 100:1, preferably 5:1 to 30:1. In another example, the weight ratio of the rubber to the regenerated rubber is 5:1 to 25:1 or 15:1 to 25:1.

[0023] In another example of aspect 1, the regenerated rubber slurry and the rubber mixture are blended in a vessel with agitation for at least 10 minutes, for example at least 10 to 120

minutes, and at a temperature in the range of 15-35 °C or 20-55 °C and/or at a pressure in the range of 2.75 to 115 psia. In an example, the vessel is a continuous stirred tank reactor.

[0024] In another example of aspect 1, a portion of the first solvent and the second solvent in the rubber blend mixture is removed with a desolventizer, for example, the solvent is removed at a temperature in the range of 90-130 °C. In an example, the desolventization can be accomplished by drum drying, extruder drying, vacuum drying, and spray drying.

[0025] In another example of aspect 1, the portion of the first solvent and the second solvent in the rubber blend mixture is removed under a vacuum in the range of -750 to 0 mmHg, or -50 to 0 mmHg, for example, in the desolventizer.

[0026] In another example of aspect 1, the rubber blend product has less than 2.5 or less than 5 weight percent of the first solvent, the second solvent, or a combination thereof based on the total weight of the rubber blend product.

[0027] In another example of aspect 1, the rubber blend product has a total moisture content, for example the total of the first and second solvents, of less than 5, preferably less than 2.5 weight percent based on the total weight of the rubber blend product.

[0028] In another example of aspect 1, the rubber blend product includes 1-50, 4-20 or 2-8 parts by weight of the regenerated rubber for every 100 parts by weight of the rubber.

[0029] In another example of aspect 1, the rubber blend product includes portions of the regenerated rubber having a rubber matrix intertwined with portions of the rubber (e.g., non-regenerated rubber). For example, regenerated rubber particles include a matrix that has intertwined with the rubber. In another example, the rubber blend product can have a crosslinked rubber network.

[0030] In a second aspect, disclosed is a rubber product that includes a blend of regenerated rubber and natural or synthetic non-regenerated rubber. In an example, the blend of regenerated rubber and natural or synthetic non-regenerated rubber is prepared by the method of aspect 1 or any combination of the examples of aspect 1.

[0031] In an example of aspect 2, a rubber composition includes the rubber product.

[0032] In an example of aspect 2, a tire includes the rubber product or a rubber composition that includes the rubber product.

[0033] In an example of aspect 2, the rubber product or a rubber composition that includes the rubber product is included in a tire component, for example, a tread or sidewall.

[0034] The above aspects (or examples of those aspects) may be provided alone or in combination with any one or more of the examples of that aspect or another aspect discussed above; e.g., the first aspect may be provided alone or in combination with any one or more of the examples of the first aspect, second aspect, third aspect or other aspects discussed above.

[0035] Additional features and advantages will be set forth in the detailed description which follows, and in part will be readily apparent to those skilled in the art from that description or recognized by practicing the embodiments as described herein, including the detailed description which follows, the claims, as well as the appended drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

[0036] The present disclosure is better understood when the following detailed description is read with reference to the accompanying drawings.

[0037] FIG. 1 shows a process diagram for preparing a rubber blend product according to an embodiment of the present invention.

[0038] FIG. 2 shows an example illustration of non-regenerated rubber intertwined with swollen regenerated rubber according to an embodiment of the present invention.

DETAILED DESCRIPTION

[0039] The terminology as set forth herein is for description of the embodiments only and should not be construed as limiting the invention as a whole.

[0040] Herein, when a range such as 5-25 (or 5 to 25) is given, this means preferably at least or more than 5 and, separately and independently, preferably not more or less than 25. In an example, such a range defines independently 5 or more, and separately and independently, 25 or less.

[0041] The present disclosure relates to methods of preparing rubber blends that achieve a non-regenerated rubber source (e.g., natural rubber) being in direct contact with regenerated rubber and/or portions of the non-regenerated rubber source penetrating into regenerated rubber,

for instance, regenerated rubber molecules or particles. The regenerated rubber is prepared by allowing the regenerated rubber to be in contact with a fluid (e.g., solvent, organic solvent, non-aqueous solvent) for a period of time to form a swollen version of the regenerated rubber having a larger volume and open voids or expanded sections for contact with the non-regenerated rubber. The swollen regenerated rubber is brought into contact with the non-regenerated rubber source for forming a rubber blend mixture that includes the non-regenerated rubber being inter-mixed with the swollen regenerated rubber to form an intertwined rubber matrix of regenerated rubber and non-regenerated rubber. Fluids such as non-aqueous solvents can be removed from the rubber blend to form a rubber blend product of regenerated rubber and non-regenerated rubber for use in rubber compositions. The rubber compositions can be used in forming one or more components of a tire or other rubber article (e.g., hose).

[0042] For the regenerated rubber of the rubber blend, various forms of rubber can be used, for example, rubber recovered from end-of-life tires, reclaimed rubber, ground rubber, devulcanized rubber or combinations thereof. The suitable rubber species of the regenerated rubber are not limited, and may include one or more selected from a natural rubber and a synthetic rubber. Examples of a synthetic rubber, where a diene rubber is typical, include a polyisoprene rubber, a styrene-butadiene copolymer rubber, a high-cis-1,4-polybutadiene rubber, a low-cis-1,4-polybutadiene rubber, an ethylene-propylene-diene terpolymer, a chloroprene rubber, a butyl rubber, a halogenated butyl rubber, an acrylonitrile-butadiene rubber, and combinations thereof. In one or more embodiments, a reclaimed rubber can be used and can be a powdered rubber or scum rubber, which is a vulcanized powdered rubber recycled from waste rubber products (reclaimed powdered rubber).

[0043] In other embodiments, a recycled particulate rubber can be used as the regenerated rubber. Recycled particulate rubber is typically broken down and reclaimed and/or recycled by any number of processes, which can include mechanical / physical breakdown, grinding, chemical breakdown, devulcanization, grinding (e.g., cryogenic), a combination thereof, etc. The term recycled particulate rubber can relate to both vulcanized and devulcanized rubber, where devulcanized recycle or recycled rubber relates to rubber which has been vulcanized, ground into particulates and may have further undergone substantial or partial devulcanization. For instance, the recycled particulate rubber used is essentially free of devulcanization. In a situation where the vulcanized rubber contains wire or textile fiber reinforcement, such wire or reinforcement

can be removed by any suitable process such as magnetic separation, air aspiration and/or air flotation step. In some examples, the recycled particulate rubber includes cured, i.e., vulcanized (cross-linked) rubber that has been ground or pulverized into particulate matter having a mean average particle size.

[0044] In one or more embodiments, the regenerated rubber may be in particulate form such that the rubber particulates have a mean average particle size of about 35 to about 600 microns, including 35 to 600 microns, about 100 to about 350 microns, 100 to 350 microns, about 150 to about 250 microns, and 150 to 250 microns. The average particle size may be measured by any conventional means known in the art, for instance, methods according to ASTM D5644. In certain embodiments, the regenerated rubber may correlate to a U.S. mesh size of about 400 to about 25, including 400 to 25, about 300 to about 100, 300 to 100, about 250 to about 150, and 250 to 150.

[0045] The regenerated rubber is blended with the non-regenerated rubber (e.g., natural rubber) for forming the rubber blend mixture. For mixing the two rubber components, the regenerated rubber may be provided with one or more solvents. The one or more solvents can be an organic solvent. In one or more embodiments, the solvents can be a hydrocarbon solvent, which includes non-polar and polar hydrocarbon solvents and combinations thereof. The non-polar hydrocarbon solvent may include C₅ to C₁₀ straight chain hydrocarbons, C₅ to C₁₀ branched chain hydrocarbons, C₅ to C₁₀ cyclic hydrocarbons, C₆ to C₁₀ aromatic hydrocarbons, and mixtures thereof. In one or more embodiments, the solvents used to prepare the rubber blend mixture are non-aqueous such that the rubber blend mixture is free of or substantially free of water.

[0046] In other embodiments, the non-polar hydrocarbon solvent includes a pentane such as cyclopentane, n-pentane, iso-pentane, neo-pentane, and mixtures thereof. Yet in other embodiments, the non-polar hydrocarbon solvent includes a hexane such as n-hexane, iso-hexane, 3-methylpentane, 2,3-dimethylbutane, neo-hexane, cyclohexane, and mixtures thereof. In these or other embodiments, the non-polar hydrocarbon solvent includes a C₆ to C₁₀ aromatic hydrocarbon such as benzene, toluene, o-xylene, m-xylene, p-xylene, ethylbenzene, 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, mesitylene, 2-ethyltoluene, 3-ethyltoluene, 4-ethyltoluene, and mixtures thereof. In these or other embodiments, the hydrocarbon solvent includes a polar organic solvent such as acetone, C₁-C₄ alcohols, C₂-C₄ diols, propane, n-butane,

isobutane, propene, 1-butene, isobutene, trans-2-butene, cis-2-butene, 1-pentene and mixtures thereof.

[0047] The rubber source for the non-regenerated rubber can be at least one of natural rubber, disclosed below, and synthetic diene-based rubbers. As the synthetic diene-based rubber is preferable one synthesized through emulsion polymerization or solution polymerization. As the synthetic diene-based rubber are concretely mentioned polyisoprene rubber, styrene-butadiene copolymer rubber, polybutadiene rubber, ethylene-propylene-diene rubber, chloroprene rubber, halogenated butyl rubber, acrylonitrile-butadiene rubber and so on.

[0048] As a natural rubber source, any suitable natural rubber from a sustainable source may be used for mixing with the regenerated rubber. In one example, a natural polyisoprene may be used. As used herein, the term “natural polyisoprene” refers to a polymer consisting essentially of cis-1,4-polyisoprene. Pure cis-1,4-polyisoprene is found in various trees, shrubs and plants, e.g., *Hevea brasiliensis*, (i.e., the Amazonian rubber tree), *Castilla elastica* (i.e., the Panama rubber tree), various *Landophia* vines (*L. kirkii*, *L. heudelotii*, and *L. owariensis*), various dandelions (i.e., *Taraxacum* species of plants), and *Parthenium argentatum* (guayule shrubs). Although the present disclosure focuses on guayule as the source of the cis-1,4-polyisoprene used in various embodiments of a rubber blend method herein, the processes disclosed herein should not be viewed as being limited to only guayule as the source for cis-1,4-polyisoprene.

[0049] In certain embodiments, one of the naturally sourced and renewable rubbers above, such as natural rubber, including Hevea and/or guayule rubber, or natural polyisoprene is selected and consists of the entire natural rubber source for mixing with the regenerated rubber for forming the blend.

[0050] In some embodiments, the natural rubber source can be present in a cement. As used herein, the term “cement” refers to a rubber cement, which is a solution comprising natural polyisoprene dissolved in one or more organic solvents. In various embodiments, a cement may include a single organic solvent (e.g., cyclohexane, acetone), or a blend of solvents as the diluent for the natural rubber.

[0051] The non-regenerated rubber source for blending with the regenerated rubber is provided with one or more solvents, preferably a non-aqueous solvent. The one or more solvents can be an organic solvent. In one or more embodiments, the solvents can be a hydrocarbon

solvent, which includes non-polar and polar hydrocarbon solvents and combinations thereof. The non-polar hydrocarbon solvent may include C₅ to C₁₀ straight chain hydrocarbons, C₅ to C₁₀ branched chain hydrocarbons, C₅ to C₁₀ cyclic hydrocarbons, C₆ to C₁₀ aromatic hydrocarbons, and mixtures thereof. In various embodiments, particular combinations of solvents provide an azeotropic mixture, thus simplifying removal of the solvents at a later stage.

[0052] In particular embodiments, the non-polar hydrocarbon solvent includes a pentane such as cyclopentane, n-pentane, iso-pentane, neo-pentane, and mixtures thereof. In these or other embodiments, the non-polar hydrocarbon solvent includes a hexane such as n-hexane, iso-hexane, 3-methylpentane, 2,3-dimethylbutane, neo-hexane, cyclohexane, and mixtures thereof. In these or other embodiments, the non-polar hydrocarbon solvent includes a C₆ to C₁₀ aromatic hydrocarbon such as benzene, toluene, o-xylene, m-xylene, p-xylene, ethylbenzene, 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, mesitylene, 2-ethyltoluene, 3-ethyltoluene, 4-ethyltoluene, and mixtures thereof. In these or other embodiments, the hydrocarbon solvent includes a polar organic solvent such as acetone, C₁-C₄ alcohols, C₂-C₄ diols, propane, n-butane, isobutane, propene, 1-butene, isobutene, trans-2-butene, cis-2-butene, 1-pentene and mixtures thereof.

[0053] The regenerated rubber and solvent, a first solvent, is blended with the non-regenerated rubber that may be provided with another solvent, a second solvent, to form a rubber blend mixture. The first and second solvents may be the same or different, or share at least one common solvent if multiple solvents make up the first and/or second solvent. Combining the rubber components can take place in a vessel, tank or suitable container such that the rubber components can be introduced by conventional means (e.g., a transfer pump). The regenerated rubber and solvent and/or the non-regenerated rubber and solvent may be pre-mixed prior to being introduced to each other for blending.

[0054] Multiple aspects of the processes disclosed herein are conducted at a temperature or temperatures of 10-80° C. (i.e., different aspects of the process may be conducted at the same temperature or at different temperatures). For example, the regenerated rubber and solvent may be mixed in a vessel or similar suitable container to form a mixture or regenerated rubber slurry. The regenerated rubber and solvent can be mixed at any suitable temperature, for example, in a range of 15 to 55° C or at ambient or room temperature, 20 to 25° C and/or at a pressure in the range of 2.75 to 115 psia.

[0055] The regenerated rubber is present in the range of 1 to 30, 2 to 20 or 3 to 12 weight percent based on the total weight of the mixture or regenerated rubber slurry. The solvent or solvents is preferably present in the mixture or regenerated rubber slurry in the range of 70 to 99, 75 to 98 or 80 to 95 weight percent based on the total weight of the mixture. The regenerated rubber and first solvent can be mixed, preferably at low shear and/or non-homogenized conditions, for any suitable time, preferably to permit the regenerated rubber or particles or molecules thereof time to swell for incorporation into the matrix or free chains of the non-regenerated or natural rubber, for instance, 10 minutes to 2 hours, 15 minutes to 1.5 hours, 25 minutes to 1 hour, or 30 minutes, 35 minutes or 45 minutes. In one or more embodiments, the low shear or non-homogenized mixing can be carried out for 2 hours to 2 weeks, 4 hours to 1 week, or 6 hours, 12 hours, 24 hours, 48 hours or 72 hours.

[0056] The formation of the mixture or regenerated rubber slurry allows the regenerated rubber to swell in the presence of the solvent or solvents to form swollen regenerated rubber, which can be in the form of particles or be described as molecules. The use of an organic solvent, as opposed to water, allows the regenerated rubber to swell, which is generally not achievable with use of water. The swollen regenerated rubber has an increased size or volume as compared to the regenerated rubber used to form the slurry. The swelling of the regenerated rubber is believed to open the rubber to create open voids or areas for easier penetration or intermixing with the non-regenerated rubber.

[0057] The non-regenerated rubber and solvent may be mixed in a vessel or similar suitable container to form a non-regenerated rubber mixture. The non-regenerated rubber and solvent can be mixed at any suitable temperature, for example, in a range of 15 to 35° or 55° C or at ambient or room temperature, 20 to 25° C and/or at a pressure in the range of 2.75 to 115 psia. The non-regenerated rubber is present in the range of 1 to 30, 2 to 20 or 3 to 12 weight percent based on the total weight of the rubber mixture. The solvent or solvents is preferably present in the rubber mixture in the range of 70 to 99, 75 to 98 or 80 to 95 weight percent based on the total weight of the mixture.

[0058] In another example, a natural rubber-containing cement, inclusive of the polymerization solvents and natural rubber source, can be used for mixing with the regenerated rubber and solvent. The natural rubber-containing cement can include the natural rubber source present in the range of 5 to 30, 10 to 25 or 12 to 20 weight percent based on the total weight of

the cement, with the remaining weight being the primarily the polymerization solvents, for instance, at 70 to 99, 75 to 98 or 80 to 95 weight percent. In one example, the blend of polymerization solvents of the natural rubber-containing cement includes one or more of a hexane and acetone. Hexanes can include at least one of an isohexane and a cyclohexane.

[0059] During mixing to form the rubber blend mixture, the total non-regenerated or natural rubber to total regenerated rubber amounts can be present in a weight ratio in the range of 100:1 to 100:50, 100:2 to 100:30, 100:3 to 100:20 or 100:4 to 100:15.

[0060] The regenerated rubber and first solvent and non-regenerated rubber and second solvent can be mixed for any suitable time to uniformly distribute the rubber components and allow direct contact between the regenerated rubber and the non-regenerated rubber in the first and second solvents. The mixing step can be broken up into segments if desired depending on a particular process or equipment availability. In another example, the regenerated rubber and first solvent and non-regenerated rubber and second solvent can be mixed at a constant temperature or within an ambient or room temperature range before being allowed to cool, if needed, to about the range of 20° to 30° C. In yet another example, the regenerated rubber and first solvent and non-regenerated rubber and second solvent can be mixed for 25 minutes to 4 hours, 35 minutes to 2.5 hours, 45 minutes to 2 hours, or 60 minutes, 1.5 hours or 2 hours. Mixing times can be adjusted depending on the shear force applied to the rubber components. For instance, mixing time can be measured by residence time in a vessel equipped with an agitator or mixing means posited in the vessel. In another example, mixing time can be measured by the passage of the rubber components and solvents through a recycle loop that contains a mixing apparatus (e.g., a pump, static mixer, homogenizer, throttle valve, etc.), taking into account the volume of the recycle loop and the number of cycles through the loop.

[0061] In one or more embodiments, the non-regenerated regenerated rubber and first solvent and non-regenerated rubber and second solvent are mixed under low shear mixing to the rubber blend mixture. One example of low shear mixing is agitation. Agitation can be achieved by shaking or rotating a container containing the regenerated regenerated rubber and first solvent and non-regenerated rubber and second solvent.

[0062] After mixing, the rubber blend mixture can be stored for later use or transferred directly to a downstream process. A downstream process can include solvent removal to reduce

or entirely eliminate the presence of any residual polymerization solvents, first or second solvent in the rubber blend mixture.

[0063] In one or more embodiments, the rubber blend mixture can be pre-heated in a vessel or desolventizer to remove any solvent present from mixing the regenerated rubber and natural rubber. The desolventizer or vessel may include moving components for further agitating and mixing of the rubber blend mixture. Operation of the mixing step in a desolventizer can beneficially remove solvents from the mixture and reduce or eliminate downstream operations that address solvent removal of the formed rubber blend mixture. Temperature in a desolventizer can be maintained above the boiling point of the solvents but below that of the rubber components, for example, in the range of 60° to 140° C, or 80° to 130° C. The vessel or desolventizer may be equipped with an agitator or other mixing means in order to carry out the mixing step on the rubber components for forming the rubber blend mixture.

[0064] In other embodiments, the rubber blend mixture is de-solventized. Practice of these embodiments of the invention are not necessarily limited by the methods used to remove solvent from the rubber blend mixture. The skilled person will appreciate that several methods can be used to remove the solvent from the rubber blend mixture and thereby provide a de-solventized rubber blend, which may also be referred to as a rubber blend product or a dried rubber blend. In one or more embodiments, the de-solventized rubber blend product includes less than 5 wt %, in other embodiments less than 2 wt %, and in other embodiments less than 0.5 wt % solvent (e.g. less than 0.25 weight percent volatile organic compounds) based on total weight of the rubber blend product.

[0065] In one or more embodiments, the rubber blend mixture is directly desolventized, which refers to a process whereby the solvent is separated from the solids portion of the mixture (i.e. separated from the polymer and regenerated rubber) to form a product that is substantially a solid composite of the polymer and regenerated rubber. This can be distinguished from indirect desolventization methods such as steam desolventization whereby water is added to drive off the solvent and thereby produce a composition that would include water and polymers, whereas the rubber blend product is substantially free of water or devoid of water. Direct desolventization techniques, as well as the equipment for performing these methods, are generally known in the art. For example, the temperature of the rubber blend mixture can be increased or maintained at a temperature sufficient to volatilize the solvent. Also, the pressure within the vessel in which the

desolventization is conducted can be decreased, which will assist in the volatilization of solvent. Still further, the rubber blend mixture can be agitated, which may further assist in the removal of solvent from the masterbatch. In one embodiment, a combination of heat, decreased pressure, and agitation can be employed.

[0066] The rubber blend product can also be used to prepare a rubber composition, for example, a rubber composition for use as a component of a vehicle tire. The rubber composition can include other components as known in the art and can include, but are not limited to, elastomers, fillers, accelerators, cure packages, sulfur, zinc oxide, waxes, processing oils, ozone agents, antioxidants, and the like. The liquid components of the dispersion can be removed, primarily the solvents, and the remaining rubber component (e.g., particles or molecules) can be dried for incorporation into a rubber composition. The liquid components can be removed as known in the art, for instance, by oven drying, vacuum evaporation, spray drying or the like. The rubber components from the blend can be incorporated into the ingredients of the rubber composition as known in the art, for example, in a Banbury mixer, a kneader, an extruder and the like.

[0067] Turning to FIG. 1, there is shown tank 14 for forming the regenerated rubber slurry by feeding regenerated rubber 10 and one or more organic solvents 12 to tank 14. The fed components 10, 12 are mixed in tank 14 by suitable mixing equipment. The components 10, 12 are subjected to a mixing step that applies mixing and shear forces, preferably with low shear force, to form a slurry. The mixing step can be carried out in tank 14 by using a motorized mixing element mounted in the tank. A mixing element can be positioned or mounted in tank 14, or in any similarly suitable vessel, at desirable. For example, a mixing element can be top-mounted in a tank or alternatively bottom-mounted. Alternatively, the slurry can be mixed in tank 14 by transferring the components 10, 12 through a recycle loop equipped with a mixer. The recycle loop is in fluid communication with tank 16 and reintroduced the mixed slurry back into tank 14 after passing through a mixer mounted in-line in the recycle loop.

[0068] The regenerated rubber of stream 10 is preferably mixed sufficiently in solvent 12 to swell such that the slurry 16 contains swollen regenerated rubber. Alternatively, in addition to or separately the regenerated rubber of tank 14 is fed to vessel 30 and allowed to swell in the presence of the non-regenerated and/or natural rubber mixture or prior to the introduction of the non-regenerated rubber mixture to vessel 30.

[0069] For forming the non-regenerated rubber mixture, a non-regenerated or natural rubber source 20 and one or more organic solvents 22 are fed to tank 24, which can be equipped with mixing equipment for forming a mixture. The mixing equipment for tank 24 can be as described for tank 14. In other examples, a natural rubber cement including polymerization solvents can be fed to tank 14 as a natural rubber source. One or more organic solvents 22 can optionally be fed to tank 14 to be mixed with the natural rubber cement, for instance, to reduce the solid content of the natural rubber mixture to be less than that in the cement. To aid in flowability and transport, the natural rubber cement can be at a higher temperature and/or tank 14 can be equipped with a heating means, for example a steam jacket or heating coils, to maintain cement at a temperature above the solubility temperature for the natural rubber in the cement.

[0070] The regenerated rubber slurry 16 and non-regenerated rubber mixture 26 are transferred, for example by a pump, to vessel 30 that applies mixing and shear forces to the rubber components to form a rubber blend mixture 34. The mixing step can be carried out in tank 30 as shown by using a motorized mixing element 32 mounted in the tank. A mixing element 32 can be positioned or mounted in tank 30, or in any similarly suitable vessel, at desirable. During mixing, the non-regenerated rubber from the non-regenerated rubber mixture 26 directly contacts the pre-formed swollen molecules of regenerated rubber. The rubber makes intimate contact with swollen molecules of regenerated rubber and some non-regenerated rubber will penetrate the swollen regenerated rubber molecules.

[0071] In one or more embodiments, a second non-regenerated rubber, as described herein, may be added to the rubber blend mixture. The second non-regenerated rubber can be added directly to tank 30 followed by further mixing to incorporate the second non-regenerated rubber into the rubber blend mixture. The second non-regenerated rubber can be added to the rubber blend mixture in a stream that contains a second non-regenerated rubber and one or more organic solvents, wherein the solvent can be the same solvent or solvents used to form the rubber blend mixture. In another example, the second non-regenerated rubber can be added to tank 30 along with non-regenerated rubber mixture 26 and regenerated rubber slurry 16 to be mixed to form the rubber blend mixture 34. In another example, the second non-regenerated rubber can be added to the rubber blend mixture downstream of tank 30, for instance, in a separate mix tank, which can be upstream of desolventizer 40, or in an instream mixing element such as an inline mixer.

[0072] The formed rubber blend mixture including swollen regenerated rubber intertwined with non-regenerated rubber is transferred to a holding tank for future use or downstream processing, or simply retained in tank 30 with or without constant agitation. Downstream processing can include solvent removal or direct incorporation into components of a rubber composition for tires. Tank 30 can also be equipped with heating means to promote further solvent removal or be in fluid communication with a solvent removal system to form a rubber blend product being substantially free of hydrocarbon solvents, for instance, one or more solvents used in the polymerization of the natural rubber cement.

[0073] In one or more embodiments as shown in FIG. 1, the rubber blend mixture 36 formed in vessel 30 is fed to a desolventizer 40 that further mixes the rubber blend and removes solvent in the blend to form a rubber blend product 42 of regenerated rubber, non-regenerated rubber and portions of non-regenerated rubber intertwined or in a matrix with regenerated rubber.

Temperature in a desolventizer 40 can be maintained above the boiling point of any solvents in the rubber blend mixture 36 (e.g., polymerization solvents), for example, in the range of 60° to 140° C, 80° to 120° C, or 90° to 115° C. The desolventizer 40 can also subject the rubber blend mixture 36 to a vacuum condition for removal of the one or more remaining solvents or other residual volatiles. The rubber blend mixture 36 can be exposed to a vacuum in the range of -750 to 0 mmHg.

[0074] The rubber blend product 42 substantially free of solvents can be separated and vacuum dried for further compounding in a rubber composition for tires or other rubberized items. The formed rubber blend product may have a non-regenerated or natural rubber to regenerated rubber weight ratio of 100:1 to 100:20, 100:1 to 100:15, 100:1 to 100:10 or 100:1 to 100:8. In other examples, the rubber blend produce may include regenerated rubber in a range of 1 to 20 phr, 1 to 15 phr, 1 to 10 phr or about 2, 3, 4, 5, 6, 7, 8 or 9 phr, wherein phr stands for part per hundred non-regenerated or natural rubber (e.g., guayule rubber) in the rubber blend product.

[0075] As shown in FIG. 2, the natural rubber, which can also be any non-regenerated rubber (not shown), in the rubber blend mixture is dispersed in one or more solvents such that the natural rubber contacts swollen regenerated rubber and becomes intertwined to form matrix of regenerated rubber and non-regenerated or natural rubber. As the solvent is removed from the rubber blend mixture, for example by a desolventizer, a rubber blend product forms and the

matrix of regenerated rubber and non-regenerated rubber become unswollen and are secured together for use in a rubber composition. The method of mixing natural rubber with swollen regenerated rubber improves the interactions between the matrix rubber or natural rubber and the regenerated rubber in the blend by using solution mixing technology.

EXAMPLES

[0076] The following examples illustrate specific and exemplary embodiments and/or features of the embodiments of the present disclosure. The examples are provided solely for the purposes of illustration and should not be construed as limitations of the present disclosure. Numerous variations over these specific examples are possible without departing from the spirit and scope of the presently disclosed embodiments. More specifically, the particular solvents, rubber components, rubber composition components and other ingredients utilized in the examples should not be interpreted as limiting since other such ingredients consistent with the disclosure in the Detailed Description can be utilized in substitution. That is, the particular ingredients in the compositions, as well as their respective amounts and relative amounts should be understood to apply to the more general content of the Detailed Description.

EXAMPLE 1

[0077] Regenerated rubber and styrene butadiene rubber blend mixture

[0078] Eight samples of styrene butadiene rubber (SBR) and regenerated crumb rubber or devulcanized rubber were prepared in glass bottles. Each sample was prepared with hexane solvent. The rubber contents in parts per hundred SBR of each bottle are shown below in Table 1.

[0079] Table 1

Rubber	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
SBR	100	100	100	100	100	100	100	100
40 mesh crumb rubber	15	30						

Devulcanized rubber			15	30				
80 mesh crumb rubber					15	30		
200 mesh crumb rubber							15	30

[0080] Glass bottles were filled with SBR, a specified amount of rubber and hexane. The filled bottles were placed in a water bath maintained at a temperature of 50° C. The bottles were slowly spun in the water bath over 48 hours to initiate a low shear mixing before removing the bottles from the water bath and storing at room temperature for three weeks. Bottles containing samples 1-2 and 5-8 yielded rubber blend mixtures that appear uniform and homogenous. Bottle 3 and 4 containing samples with the devulcanized rubber had visible separation of the rubber and hexane solvent with the rubber at the bottom of the bottles with clean solvent above. Samples from bottles 3 and 4 were not further used to prepare a dry rubber blend product.

[0081] Samples from bottles 1-2 and 5-8 were drum dried to prepare dry masterbatch samples for incorporation into a rubber composition. The dry samples yielded 115 or 130 parts of a dry rubber blend product (i.e. 100 parts SBR + 15 parts crumb rubber or 100 parts SBR + 30 parts crumb rubber). Rubber compositions containing the dry rubber blend products (i.e. Stocks 2, 4, 6, 8, 10, 12) were prepared in Barbender mixer and compared to the same rubber compositions without the dry rubber blend products but replaced with separate amounts of SBR (100 parts) and crumb rubber (15 or 30 parts) (i.e. Stocks 1, 3, 5, 7, 9, 11). Twelve separate rubber compositions were prepared as shown below in Tables 2 and 3. Amounts in Table 2 are in phr.

[0082] Table 2

	Stock 1	Stock 2	Stock 3	Stock 4	Stock 5	Stock 6
Sample 1		115				
Sample 2				130		
Sample 5						115
SBR	100		100		100	

40 mesh crumb rubber	15		30			
80 mesh crumb rubber					15	
Carbon Black	50	50	50	50	50	50
Processing Oil	10	10	10	10	10	10
Stearic Acid	2	2	2	2	2	2
Wax	2	2	2	2	2	2
6PPD*	1	1	1	1	1	1
Zinc Oxide	2.5	2.5	2.5	2.5	2.5	2.5
Sulfur	1.5	1.5	1.5	1.5	1.5	1.5
TBBS**	0.5	0.5	0.5	0.5	0.5	0.5
MBTS***	0.5	0.5	0.5	0.5	0.5	0.5
DPG****	0.3	0.3	0.3	0.3	0.3	0.3

* *N*¹-(4-Methylpentan-2-yl)-*N*¹-phenylbenzene-1,4-diamine

** N-Tertiarybutyl-2-benzothiazole sulfenamide

*** 2,2'-Dibenzothiazole Disulfide

**** N,N'-Diphenylguanidine

[0083] Table 3

	Stock 7	Stock 8	Stock 9	Stock 10	Stock 11	Stock 12
Sample 6		130				
Sample 7				115		
Sample 8						130
SBR	100		100		100	

80 mesh crumb rubber	30					
200 mesh crumb rubber			15		30	
Carbon Black	50	50	50	50	50	50
Processing Oil	10	10	10	10	10	10
Stearic Acid	2	2	2	2	2	2
Wax	2	2	2	2	2	2
6PPD*	1	1	1	1	1	1
Zinc Oxide	2.5	2.5	2.5	2.5	2.5	2.5
Sulfur	1.5	1.5	1.5	1.5	1.5	1.5
TBBS**	0.5	0.5	0.5	0.5	0.5	0.5
MBTS***	0.5	0.5	0.5	0.5	0.5	0.5
DPG****	0.3	0.3	0.3	0.3	0.3	0.3

* *N*¹-(4-Methylpentan-2-yl)-*N*¹-phenylbenzene-1,4-diamine

** N-Tertiarybutyl-2-benzothiazole sulfenamide

*** 2,2'-Dibenzothiazole Disulfide

**** N,N'-Diphenylguanidine

[0084] The rheometry of the compositions (Stocks 1-12) was measured according to ASTM D5289 (ML) and ASTM D2084 (MH). The results of the measurements are shown below in Table 4.

[0085] Table 4

	ML (dNm)	MH (dNm)
Stock 1	2.07	16.34
Stock 2	2.39	16.26

Stock 3	2.51	15.60
Stock 4	2.60	15.60
Stock 5	2.05	15.81
Stock 6	2.25	16.89
Stock 7	2.38	14.2
Stock 8	2.65	16.86
Stock 9	2.25	15.83
Stock 10	2.18	16.53
Stock 11	2.53	15.32
Stock 12	2.82	16.23

[0086] As shown in Table 4, the Stocks using the prepared rubber blend products from Samples 1-2 and 5-8 generally evidence essentially the same or significantly higher ML and MH values as compared to the same composition that replaces the sample rubber blend products with the same amount of SBR and crumb rubber in a dry blended state.

[0087] For example, Stock 2 exhibited a 15.5% increase in ML, Stock 4 exhibited a 3.6% increase in ML, Stock 6 exhibited a 9.8% increase in ML, Stock 8 exhibited a 11.3% increase in ML, and Stock 10 exhibited a 11.5% increase in ML as compared to their respective comparative Stocks having unblended SBR and crumb rubber of varying mesh sizes, namely Stocks 1, 3, 5, 7 and 11. In another example, Stock 6 exhibited a 6.8% increase in MH, Stock 8 exhibited a 18.7% increase in MH, Stock 10 exhibited a 4.4% increase in MH and Stock 12 exhibited a 5.9% increase in MH as compared to their respective comparative Stocks having unblended SBR and crumb rubber of varying mesh sizes, namely Stocks 5, 7, 9 and 11.

[0088] The trend of increasing values of ML and MH for Stocks with the solution masterbatch of Sample rubber blend products indicates a greater interaction between the SBR and crumb rubber as compared to SBR and crumb rubber being added in separate quantities. The dry mixing of the SBR and crumb rubber in Stocks 1, 3, 5, 7, 9 and 11 appears to result in no penetration or entanglement of free SBR chains with the crumb rubber.

[0089] The dynamic properties, modulus G'' , of the compositions (Stocks 1-12) were measured according to ASTM D5992, with conditions of 10 Hz, 0.25% strain and a temperature sweep from -100° C to 0° C. The results of the measurements are shown in FIGS. 3, 4 and 5.

[0090] As shown in FIG. 3, two peaks for each Stocks 2 and 4 that used SBR and Samples 1 and 2 of rubber blend products are closer than the two peaks in each Stocks 1 and 3 that used the same amount of SBR and crumb rubber in a dry blended state. Notably, the two peaks in each Stocks 2 and 4 are less distinctive compared with the two peaks for each Stocks 1 and 3. These two observations indicate more intimate interactions between SBR and crumb rubber in Stocks 2 and 4 compared to Stocks 1 and 3.

[0091] As shown in FIG. 4, two peaks for each Stocks 6 and 8 that used SBR and Samples 5 and 6 of rubber blend products are closer than the two peaks in each Stocks 5 and 7 that used the same amount of SBR and crumb rubber in a dry blended state. Notably, the two peaks in each Stocks 6 and 8 are less distinctive compared with the two peaks for each Stocks 5 and 7. These two observations indicate more intimate interactions between SBR and crumb rubber in Stocks 6 and 8 compared to Stocks 5 and 7.

[0092] As shown in FIG. 5, two peaks for each Stocks 10 and 12 that used SBR and Samples 7 and 8 of rubber blend products are closer than the two peaks in each Stocks 9 and 11 that used the same amount of SBR and crumb rubber in a dry blended state. Notably, the two peaks in each Stocks 10 and 12 are less distinctive compared with the two peaks for each Stocks 9 and 11. These two observations indicate more intimate interactions between SBR and crumb rubber in Stocks 10 and 12 compared to Stocks 9 and 11.

[0093] The dynamic properties, modulus G' , of the compositions (Stocks 1-12) were measured according to ASTM D5992, with conditions of 10 Hz, temperature 60° C, and strain sweep from 0.1% to 16%. The results of the measurements are shown in FIGS. 6, 7 and 8.

[0094] As shown in FIG. 6, the G' modulus curve was shifted upward to higher values for Stocks 2 and 4 that used Samples 1 and 2 of rubber blend products as compared to Stocks 1 and 3 that used the same amount of SBR and crumb rubber in a dry blended state. It is indicated in FIG. 6 that Stocks 2 and 4 exhibit an increased G' property and show more intimate interactions between SBR and crumb rubber in the rubber blend product of Samples 1 and 2 as compared to Stocks 1 and 3.

[0095] As shown in FIG. 7, the G' modulus curve was shifted upward to higher values for Stocks 6 and 8 that used Samples 5 and 6 of rubber blend products as compared to Stocks 5 and 7 that used the same amount of SBR and crumb rubber in a dry blended state. It is indicated in FIG. 7 that Stocks 6 and 8 exhibit an increased G' property and show more intimate interactions

between SBR and crumb rubber in the rubber blend product of Samples 5 and 6 as compared to Stocks 5 and 7.

[0096] As shown in FIG. 8, the G' modulus curve was shifted upward to higher values for Stocks 10 and 12 that used Samples 7 and 8 of rubber blend products as compared to Stocks 9 and 11 that used the same about of SBR and crumb rubber in a dry blended state. It is indicated in FIG. 8 that Stocks 10 and 12 exhibit an increased G' property and show more intimate interactions between SBR and crumb rubber in the rubber blend product of Samples 7 and 8 as compared to Stocks 9 and 11.

EXAMPLE 2

[0097] High shear mixed regenerated rubber and styrene butadiene rubber blend mixture

[0098] Two samples of styrene butadiene rubber (SBR) and regenerated crumb rubber (80 mesh) were prepared in a container and allowed to soak for one week at room temperature. Each sample was prepared with hexane solvent. The rubber contents in parts per hundred SBR of each bottle are shown below in Table 5.

[0099] Table 5

Rubber	Sample 9	Sample 10
SBR	100	100
80 mesh crumb rubber	15	30

[00100] After soaking for one week, the contents of each container were mixed for 10 minutes at 10,500-12,000 rpm using a tabletop homogenizer to achieve high shear mixing. The contents of each container were dried at 50° C for 4 hours and at -30 in H2O vacuum.

[00101] Dry samples 9 and 10 yielded 115 or 130 parts of a dry rubber blend product (i.e. 100 parts SBR + 15 parts crumb rubber or 100 parts SBR + 30 parts crumb rubber). Rubber compositions containing the dry rubber blend products (i.e. Stocks 14, 16) were prepared in Barbender mixer and compared to the same rubber compositions without the dry rubber blend products but replaced with separate amounts of SBR (100 parts) and crumb rubber (15 or 30 parts) (i.e. Stocks 13, 15). Four separate rubber compositions were prepared as shown below in Table 6. Amounts in Table 6 are in phr.

[00102] Table 6

	Stock 13 (Dry-RK008 15)	Stock 14 (Wet-RK008 15)	Stock 15 (Dry-RK008 30)	Stock 16 (Wet-RK008 30)
Sample 9		115		
Sample 10				130
SBR	100		100	
80 mesh crumb rubber	15		30	
Carbon Black	50	50	50	50
Processing Oil	10	10	10	10
Stearic Acid	2	2	2	2
Wax	2	2	2	2
6PPD*	1	1	1	1
Zinc Oxide	2.5	2.5	2.5	2.5
Sulfur	1.5	1.5	1.5	1.5
TBBS**	0.5	0.5	0.5	0.5
MBTS***	0.5	0.5	0.5	0.5
DPG****	0.3	0.3	0.3	0.3

* *N*¹-(4-Methylpentan-2-yl)-*N*⁴-phenylbenzene-1,4-diamine

** N-Tertiarybutyl-2-benzothiazole sulfenamide

*** 2,2'-Dibenzothiazole Disulfide

**** N,N'-Diphenylguanidine

[00103] The dynamic properties, modulus G'' , of the compositions (Stocks 13-16) were measured according to ASTM D5992, with conditions of 10 Hz, 0.25% strain and a temperature sweep from -100° C to 0° C. The results of the measurements are shown in FIG. 9.

[00104] As shown in FIG. 9, the curves for Stocks 13 and 14 and curves for Stocks 15 and 16 are almost identical. There is no signs of more intimate interactions between SBR and crumb rubber in the rubber blend product of Samples 9 and 10 as compared to Stocks 13 and 15. It is believed that the high shear mixing used to prepare Samples 9 and 10 did not result in the same

intimate interactions between SBR and crumb rubber as the low shear mixing carried out in Example 1.

[00105] The tensile properties, stress (MPa), of the compositions (Stocks 13-16) were measured according to ASTM D412. The results of the measurements are shown in FIG. 10.

[00106] As shown in FIG. 10, the top line in the chart is for Stock 13 and thus there was no shift upward in tensile values for Stocks 14 and 16. FIG. 10 indicates that Stocks 14 and 16 did not exhibit more intimate interactions between SBR and crumb rubber in the rubber blend product of Samples 9 and 10 as compared to Stocks 13 and 15. It is believed that the high shear mixing used to prepare Samples 9 and 10 did not result in the same intimate interactions between SBR and crumb rubber as the low shear mixing carried out in Example 1.

[00107] While various aspects and embodiments of the compositions and methods have been disclosed herein, other aspects and embodiments will be apparent to those skilled in the art. The various aspects and embodiments disclosed herein are for purposes of illustration and are not intended to be limiting, with the true scope and spirit being indicated by the claims.

CLAIMS:

1. A method for preparing a rubber blend, the method comprising:
 - a. mixing a regenerated rubber and a first solvent to form a regenerated rubber slurry;
 - b. mixing a rubber and a second solvent to form a rubber mixture;
 - c. blending the regenerated rubber slurry and the rubber mixture to form a rubber blend mixture;
 - d. removing a portion of the first solvent and the second solvent from the rubber blend mixture to form a rubber blend product.
2. The method of claim 1, wherein the regenerated rubber slurry comprises the regenerated rubber in a solids content in the range of 1-15 weight percent based on the total weight of the regenerated rubber slurry.
3. The method of claim 1, wherein the regenerated rubber in the regenerated rubber slurry swells in the first solvent to form swollen regenerated rubber.
4. The method of claim 3, wherein the swollen regenerated rubber has a larger volume as compared to the regenerated rubber used to mix with the first solvent for forming the regenerated rubber slurry.
5. The method of claim 1, wherein the rubber in the second solvent is a natural or non-regenerated rubber of the rubber mixture and penetrates the swollen regenerated rubber of the regenerated rubber slurry during the blending of step (c).
6. The method of claim 5, wherein the natural rubber is derived from a guayule plant or a guayule natural rubber cement.

7. The method of claim 1, wherein the rubber mixture comprises the rubber in a solids content in the range of 5-30 weight percent based on the total weight of the rubber mixture.
8. The method of claim 1, wherein the rubber mixture of step (b) is formed at a temperature in the range of 15-35 °C and the regenerated rubber slurry of step (a) is formed at a temperature in the range of 15-35 °C.
9. The method of claim 1, wherein the first solvent comprises a hexane, the second solvent comprises a hexane and/or the second solvent comprises an acetone.
10. The method of claim 1, wherein the rubber blend mixture comprises a weight ratio of the rubber to the regenerated rubber of 2:1 to 100:1, preferably 5:1 to 30:1.
11. The method of claim 1, wherein the rubber blend mixture comprises a weight ratio of the rubber to the regenerated rubber of 5:1 to 25:1.
12. The method of claim 1, wherein the regenerated rubber slurry and the rubber mixture are blended in a vessel with agitation for at least 10 minutes, preferably at least 10 to 120 minutes, at a temperature in the range of 10-55°C or 15-35 °C.
13. The method of claim 1, wherein the portion of the first solvent and the second solvent in the rubber blend mixture is removed with a desolventizer.
14. The method of claim 13, wherein the portion of the first solvent and the second solvent in the rubber blend mixture is removed at a temperature in the range of 90-130 °C.
15. The method of claim 1, wherein the portion of the first solvent and the second solvent in the rubber blend mixture is removed under a vacuum in the range of -750 to 0 mmHg.

16. The method of claim 1, wherein the rubber blend product comprises less than 5 weight percent of the first solvent, the second solvent, or a combination thereof based on the total weight of the rubber blend product.
17. The method of claim 1, wherein the rubber blend product comprises a total moisture content of less than 5 weight percent, preferably less than 2.5 weight percent, based on the total weight of the rubber blend product.
18. The method of claim 1, wherein the rubber blend product comprises 1-50, preferably 4-20 parts by weight of the regenerated rubber for every 100 parts by weight of the rubber.
19. The method of claim 1, wherein the rubber blend product comprises portions of the regenerated rubber having a rubber matrix intertwined with portions of the rubber.
20. The method of claim 1, wherein the first and/or second solvent is a non-aqueous solvent.

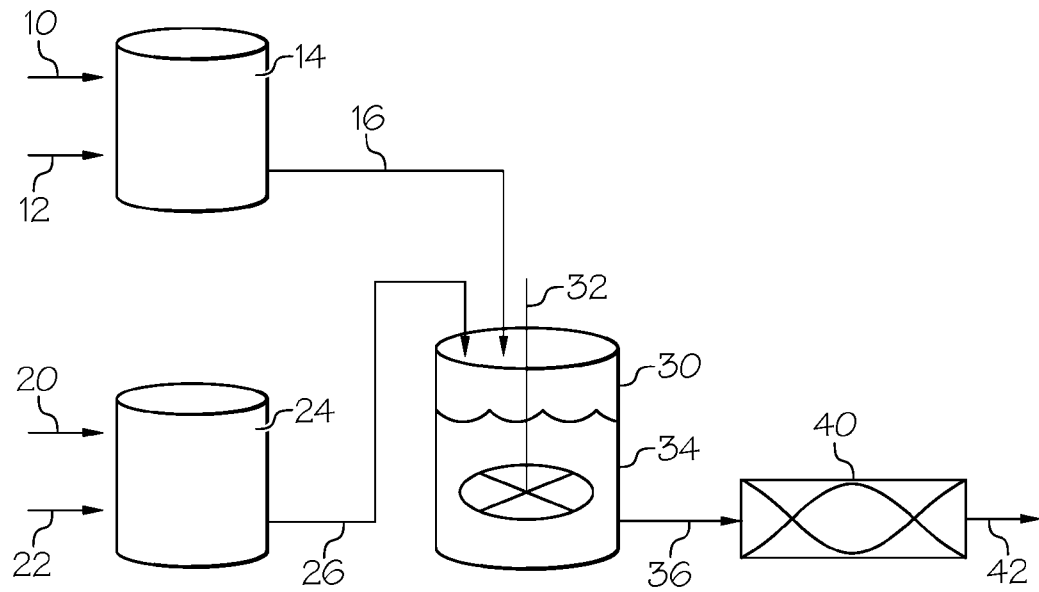


FIG. 1

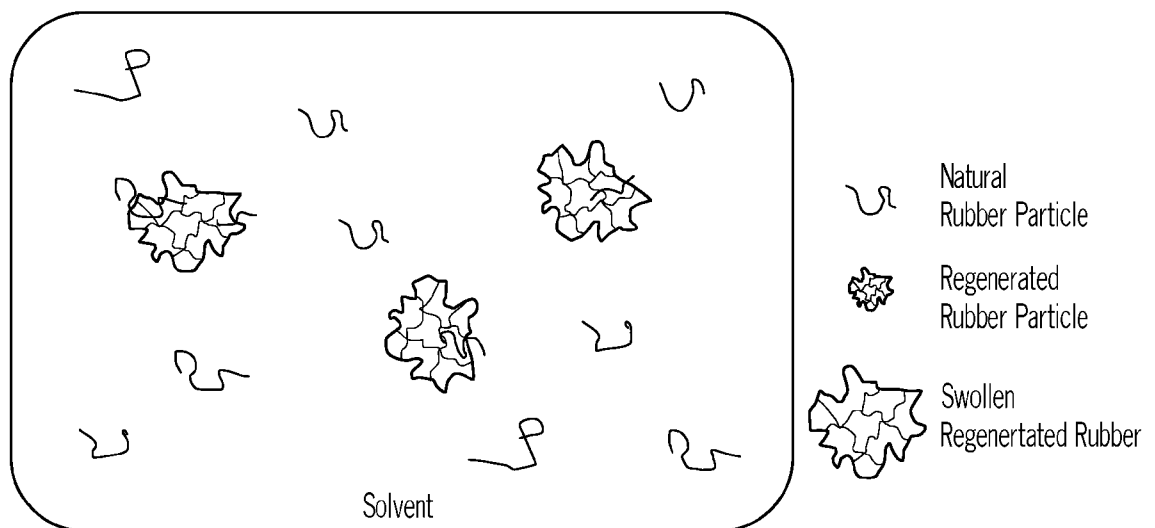


FIG. 2

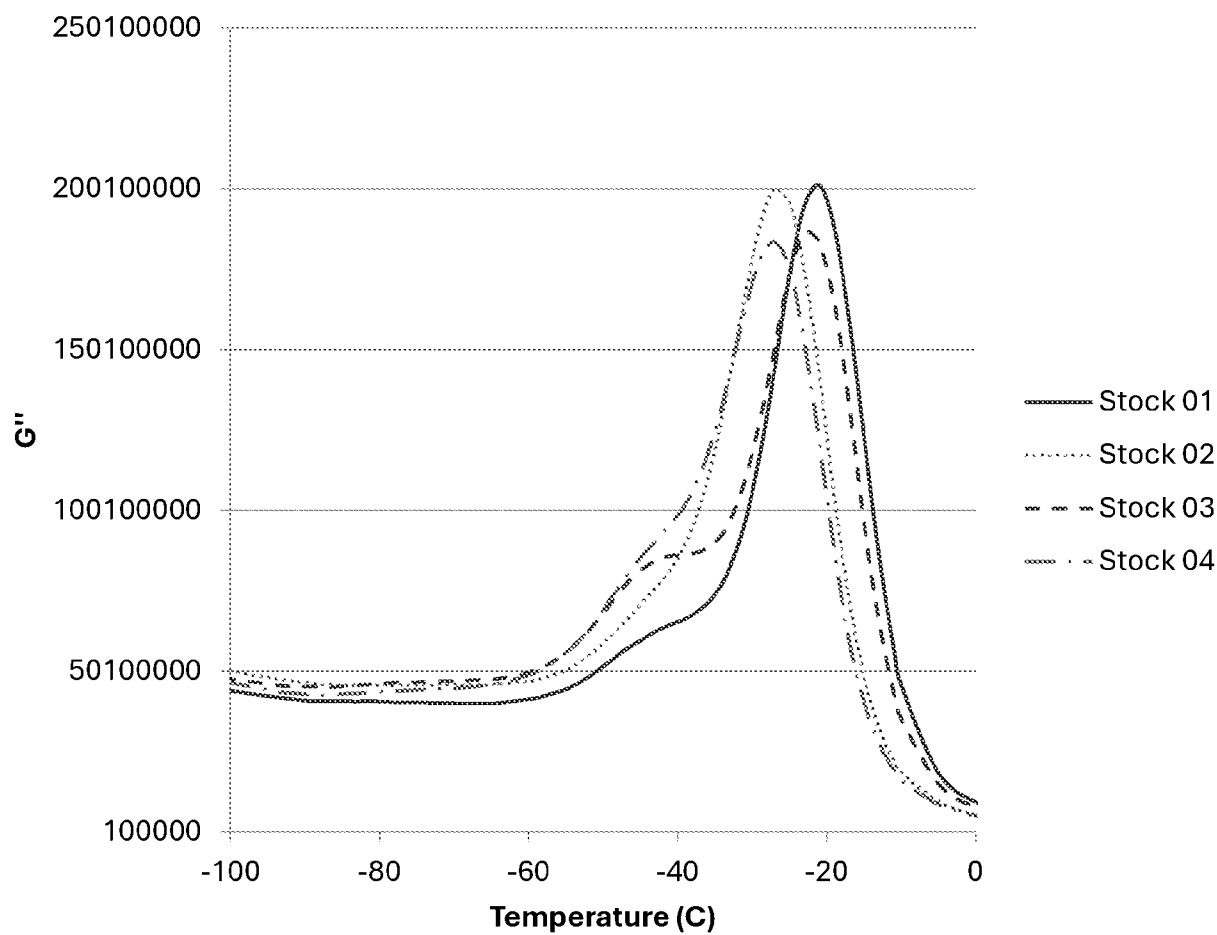


FIG. 3

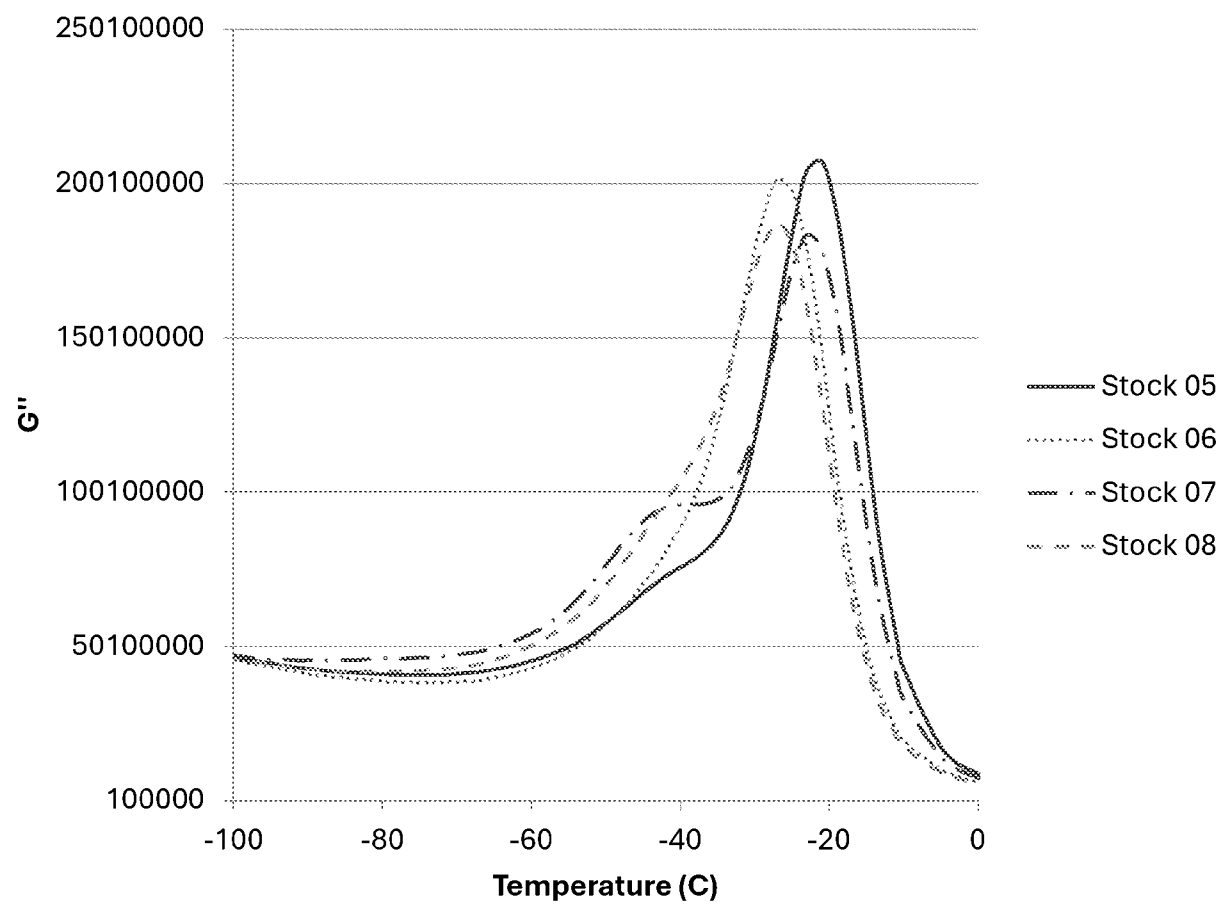


FIG. 4

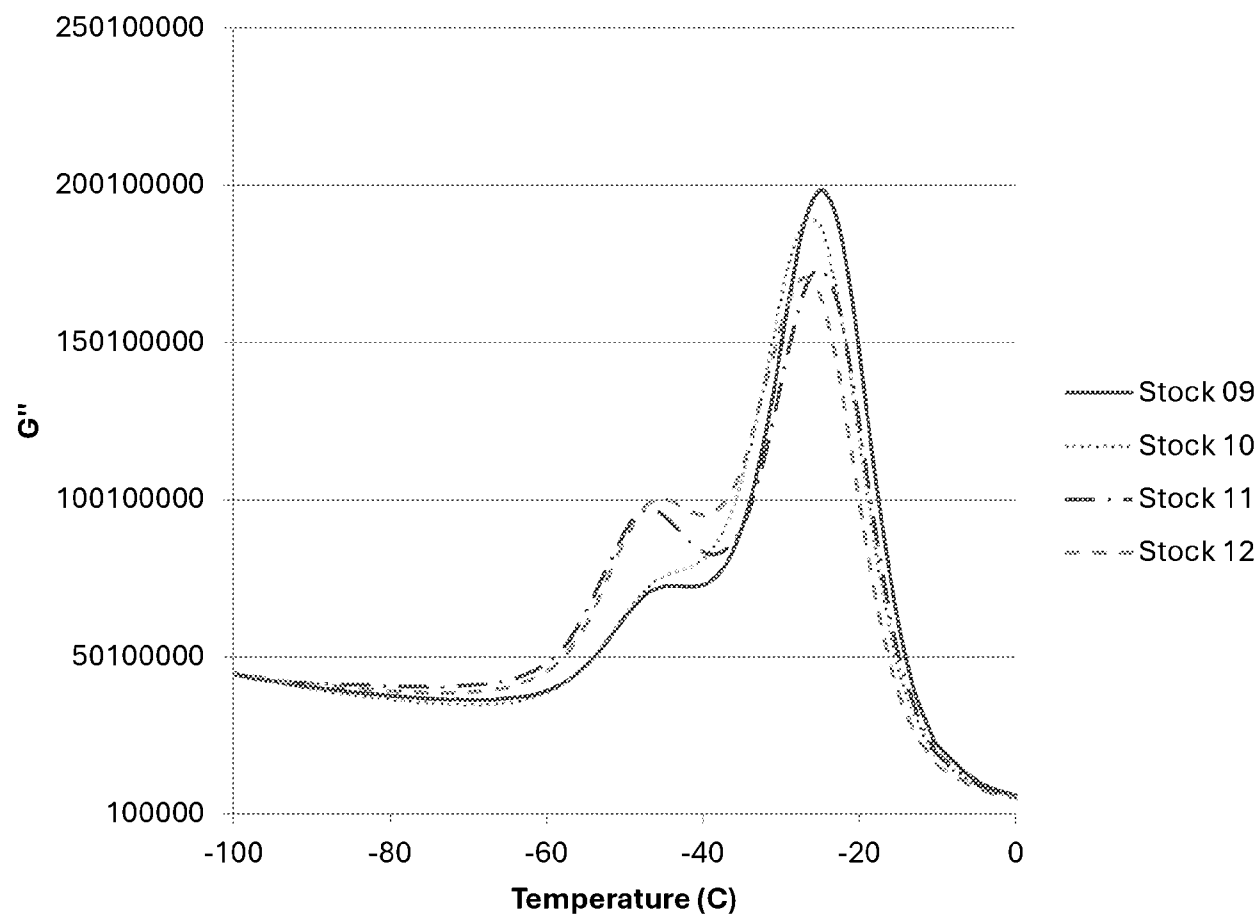


FIG. 5

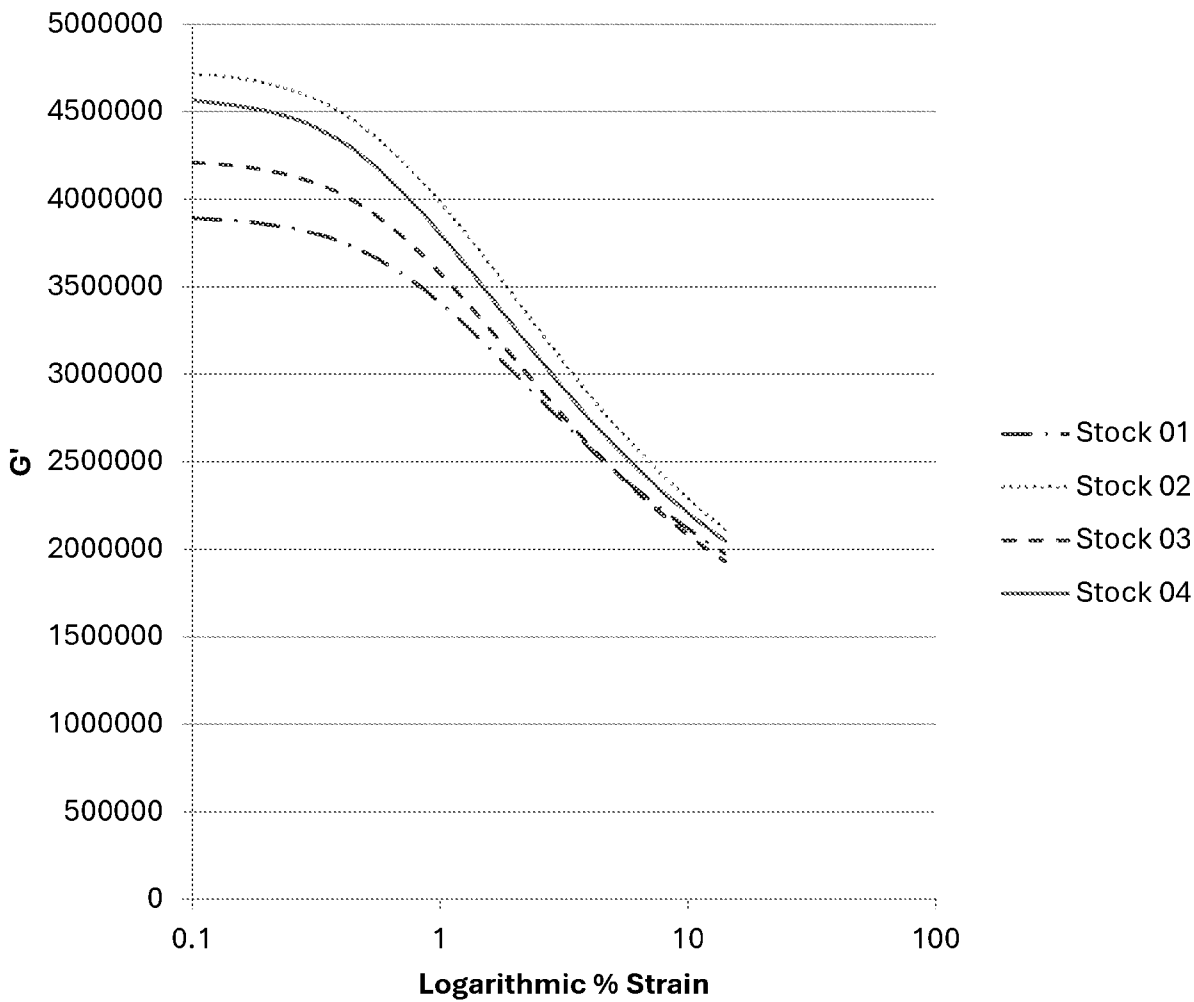


FIG. 6

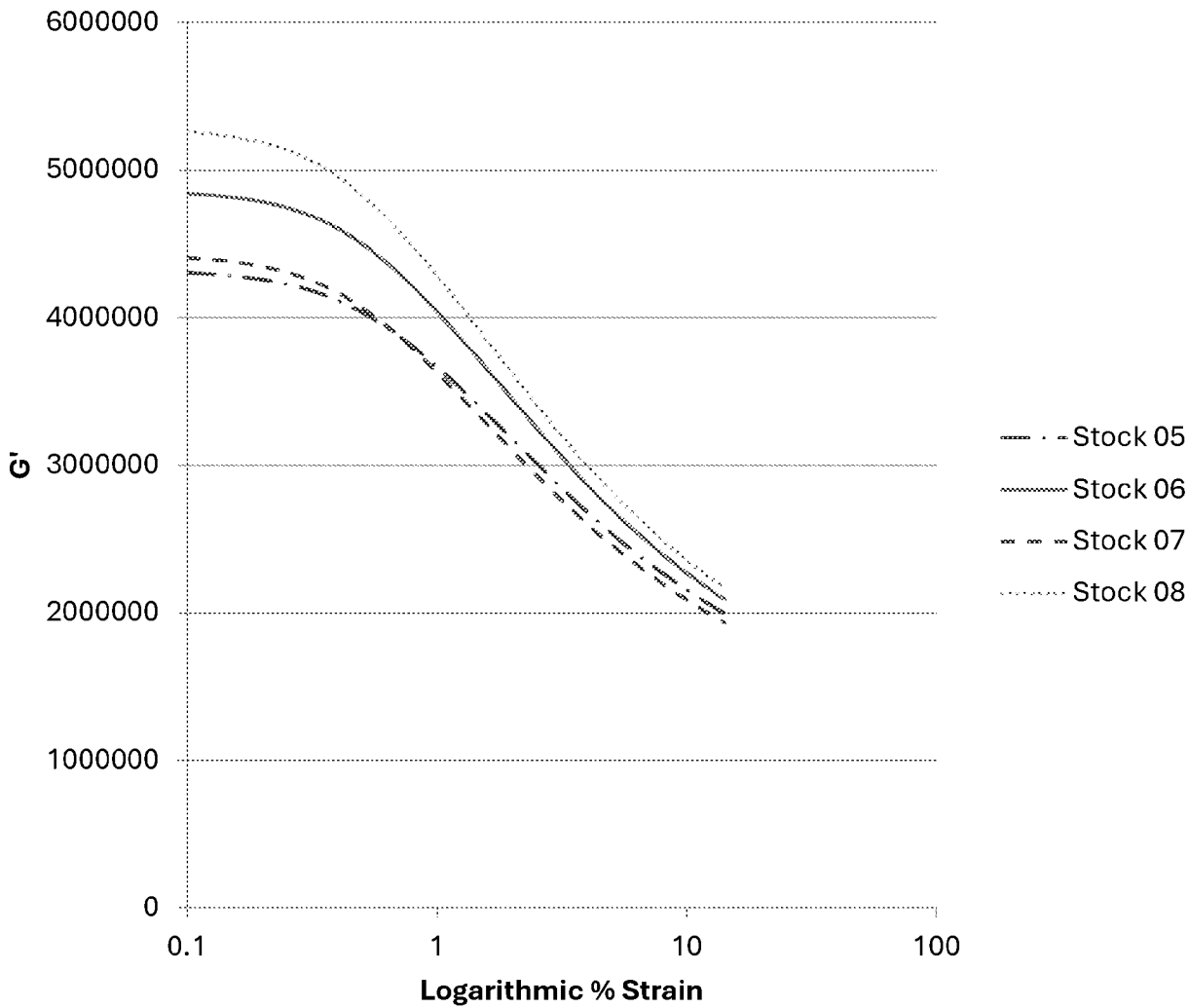


FIG. 7

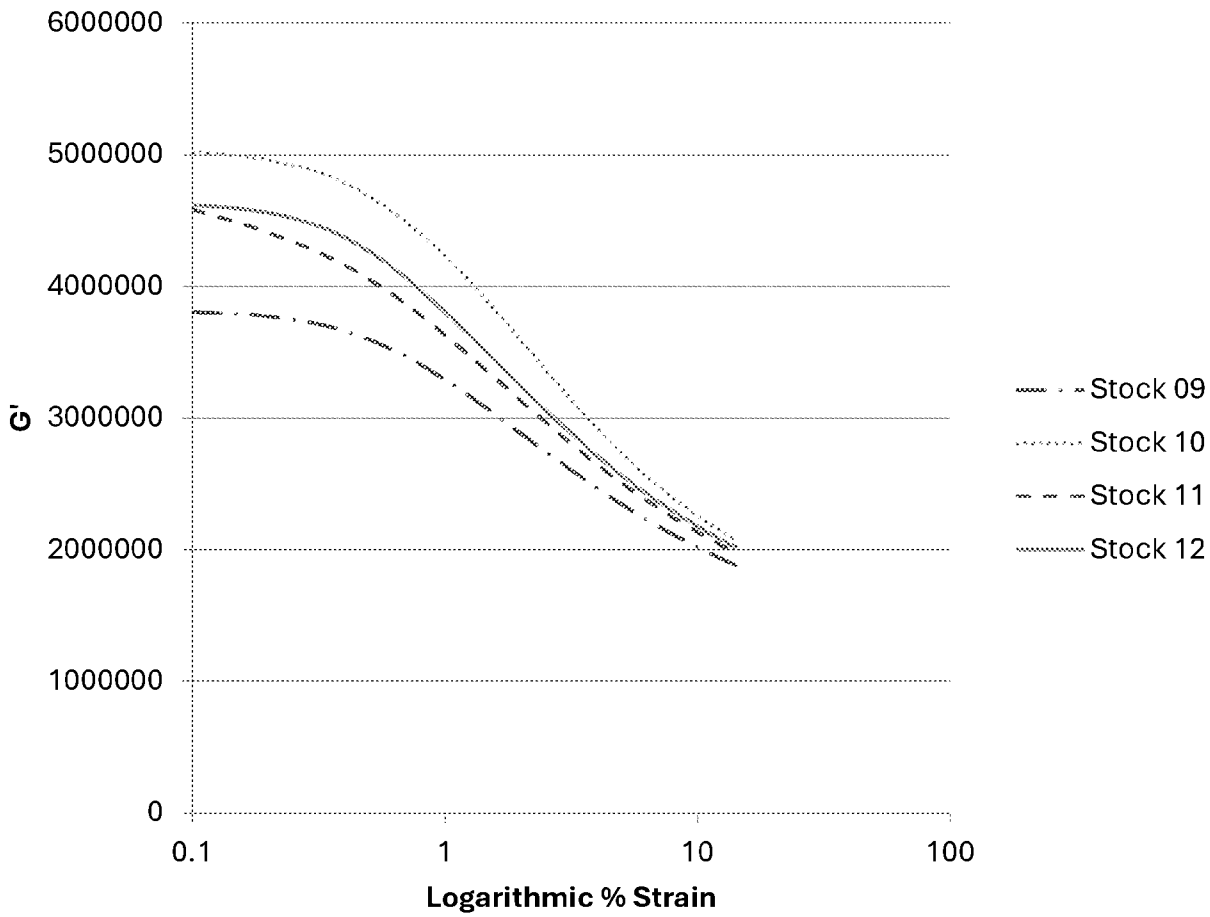


FIG. 8

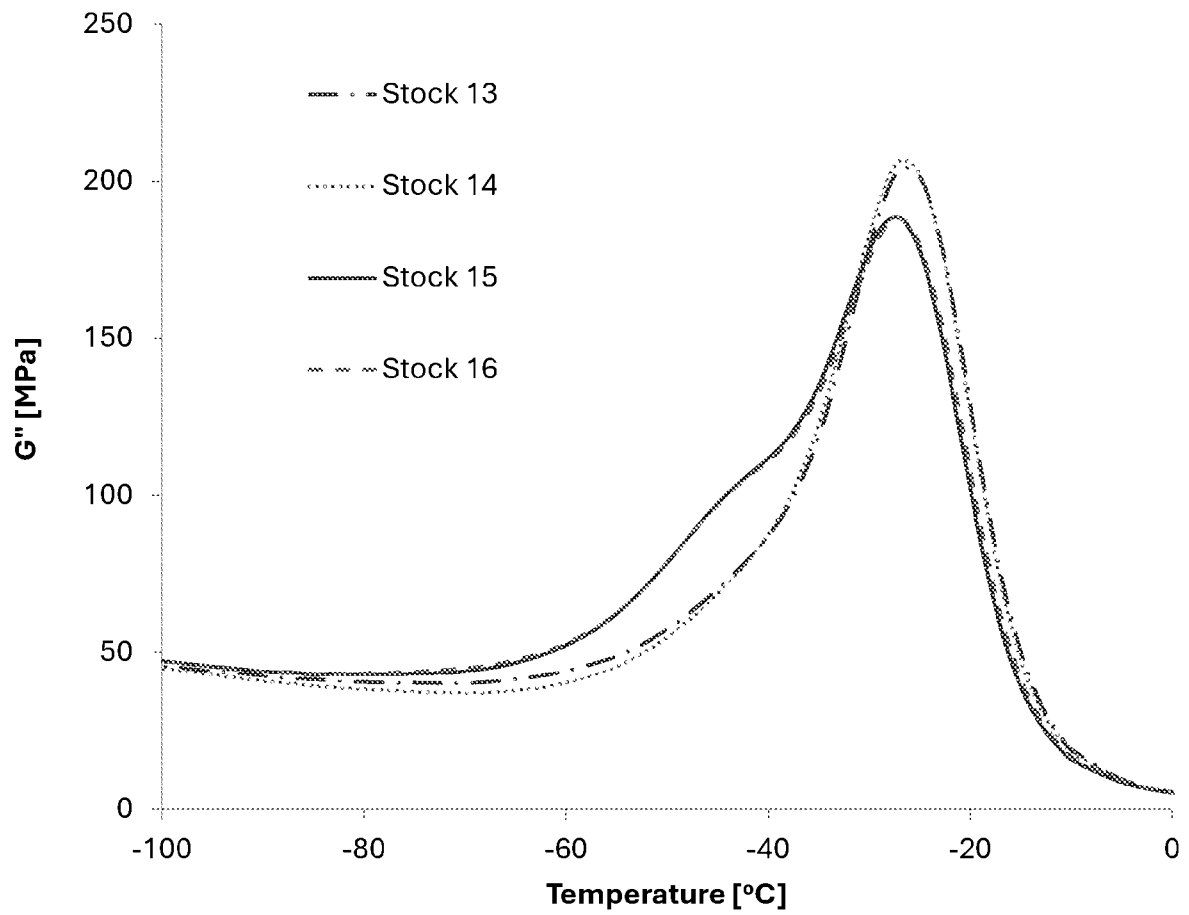


FIG. 9

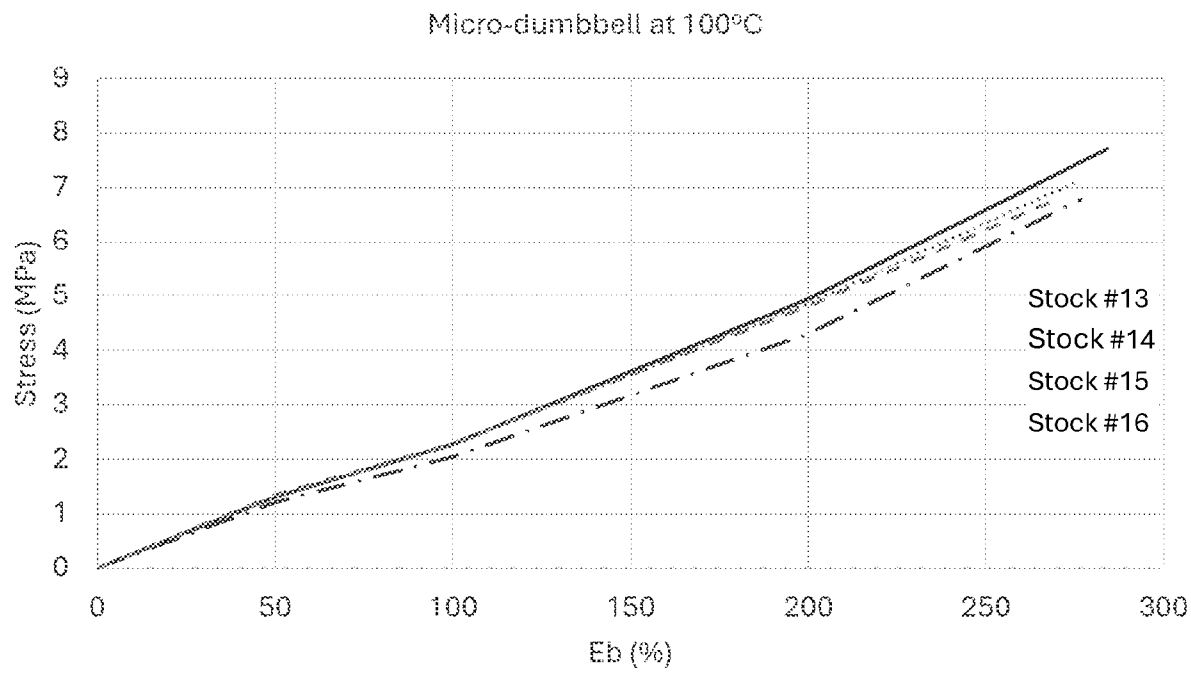


FIG. 10

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US2024/062069

A. CLASSIFICATION OF SUBJECT MATTER**C08J 3/00**(2006.01)i; **C08J 7/02**(2006.01)i; **C08L 7/00**(2006.01)i; **C08L 21/00**(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C08J 3/00(2006.01); B60C 15/00(2006.01); C08C 2/06(2006.01); C08J 11/10(2006.01); C08J 11/28(2006.01);
C08L 19/00(2006.01); C08L 23/28(2006.01); C08L 7/00(2006.01); C08L 9/06(2006.01)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Korean utility models and applications for utility models
Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

eKOMPASS(KIPO internal) & Keywords: rubber blend, regenerated rubber, solvent

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2016-0152805 A1 (JASIUNAS, C. A. et al.) 02 June 2016 (2016-06-02) abstract; claim 1; paragraphs [0037]-[0069]	1-20
Y	CN 111647198 A (BEIJING KLINTYRE ENVIRONMENTAL PROTECTION TECHNOLOGY CO., LTD.) 11 September 2020 (2020-09-11) abstract; claims 1-10	1-20
Y	CN 103435857 B (SHANDONG YONGTAI CHEMICAL GROUP CO., LTD.) 09 December 2015 (2015-12-09) claims 1-7	6
A	US 04161464 A (NICHOLAS, P. P.) 17 July 1979 (1979-07-17) claims 1-19	1-20
A	CN 101218287 A (ALBERTA RES COUNCIL) 09 July 2008 (2008-07-09) claims 1-43	1-20



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

“A” document defining the general state of the art which is not considered to be of particular relevance

“D” document cited by the applicant in the international application

“E” earlier application or patent but published on or after the international filing date

“L” document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

“O” document referring to an oral disclosure, use, exhibition or other means

“P” document published prior to the international filing date but later than the priority date claimed

“T” later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

“X” document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

“Y” document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

“&” document member of the same patent family

Date of the actual completion of the international search

18 April 2025

Date of mailing of the international search report

18 April 2025

Name and mailing address of the ISA/KR

**Korean Intellectual Property Office
189 Cheongsa-ro, Seo-gu, Daejeon
35208, Republic of Korea**

Facsimile No. +82-42-481-8578

Authorized officer

HEO, Joo Hyung

Telephone No. +82-42-481-5373

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/US2024/062069

Patent document cited in search report			Publication date (day/month/year)	Patent family member(s)			Publication date (day/month/year)
US	2016-0152805	A1	02 June 2016	US	9598564	B2	21 March 2017
CN	111647198	A	11 September 2020	None			
CN	103435857	B	09 December 2015	CN	102364466	A	29 February 2012
				CN	102850525	A	02 January 2013
				CN	102850525	B	23 September 2015
				CN	103435857	A	11 December 2013
				CN	200989298	Y	12 December 2007
US	04161464	A	17 July 1979	None			
CN	101218287	A	09 July 2008	CA	2589307	A1	01 June 2006
				CA	2589307	C	06 August 2013
				CN	101218287	B	04 January 2012
				US	2006-0116431	A1	01 June 2006
				US	7425584	B2	16 September 2008
				WO	2006-056072	A1	01 June 2006