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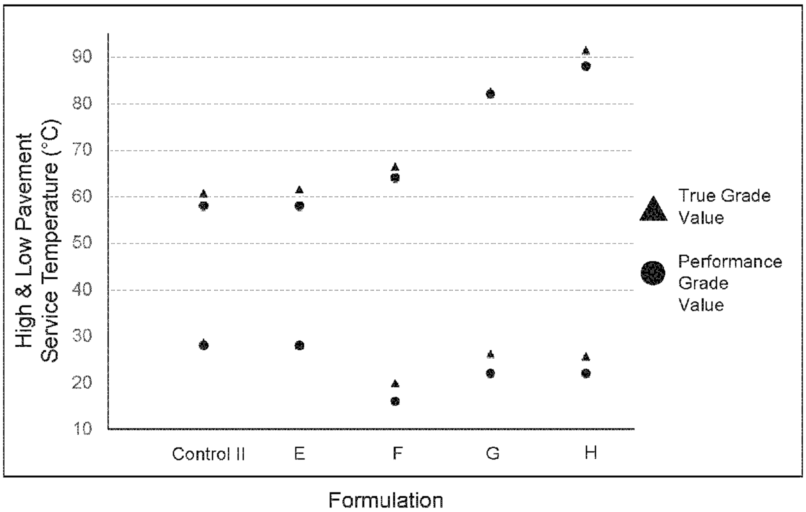

FIG. 3

(57) Abstract: Asphalt can be modified by polymers, oligomers, and waxes made from polymeric material. The addition of polymer, oligomer, or wax can increase the softening point of the asphalt, decrease the penetration of the asphalt, and/or shorten the oxidation of the asphalt. In some embodiments, polymer, oligomer, or wax is added to an oxidized asphalt. The polymer, oligomer, or wax can be made by catalytic depolymerization and/or thermal degradation of polymeric material. The polymeric material can be polystyrene, polypropylene, polyethylene, a combination of polypropylene and polyethylene or recycled plastics. In some embodiments, addition of the polymer, oligomer, or wax improves the performance grade of a paving asphalt binder alone or in combination with other modifiers such as ground tire rubber and polymers. The addition of wax can increase the high service temperature of the asphalt binder.

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MODIFICATION OF ASPHALT OXIDATION AND BINDERS WITH POLYMER WAXES

Cross-Reference to Related Applications

[0001] This application is related to and claims priority benefits from U.S. Provisional Application Serial No. 62/679,150 filed on June 1, 2018 entitled "Modification of Asphalt Oxidation with Waxes Derived from Depolymerization of Plastic". This application is also related to and claims priority benefits from U.S. Provisional Application Serial No. 62/681,344 filed on June 6, 2018 entitled "Modification of Asphalt Binders with Waxes to Improve Performance Grade".

[0002] The '150 and '344 provisional applications are hereby incorporated by reference herein in their entireties.

Field of the Invention

[0003] The present invention relates to a method of employing polymers, oligomers, and waxes, from now on just referred to as waxes, as additives in asphalt formulations. In some embodiments, the waxes are created via the depolymerization of polymers. In some embodiments, the addition of the wax(es) improve(s) asphalt properties including increasing the softening point and/or hardness of the asphalt. In some embodiments, the addition of the wax(es) to asphalt binders improves performance grade.

[0004] It is often advantageous for asphalt to resist flow at high temperatures and/or penetration from physical forces. Various applications including roofing and paving require relatively stable asphalt at high temperatures. For example, paving asphalt should be able to withstand high temperatures encountered in different climates. This ability to withstand high temperatures is conferred by the asphalt's resistance to flow at high temperatures measured by the softening point of the material (the temperature at which the asphalt achieves a specified degree of viscosity). Asphalts with high softening points are better suited for avoiding damage at higher temperatures.

[0005] In addition to resistance to flow, the hardness of an asphalt can be modified for particular applications. A penetration test serves as one metric to measure the hardness of asphalt. Paving asphalt is often made harder to reduce penetration from heavy forces, such as large trucks. Harder asphalts that are stable at high and low temperatures are also less likely to rut and/or crack.

[0006] Traditionally oxidation of asphalt is used to modify the softening point and/or hardness of the asphalt. However, the oxidation process is both timely and costly.

[0007] Asphalt binders can be used as a bonding coat in applications such as patching, paving and coating to promote adhesion in concrete products and coatings. A common method for determining asphalt binder quality is the Performance Graded System based on the idea that asphalt binder properties should be established by the conditions under which the binder is used. Performance grading uses a set of industry standard tests to measure the physical properties of the asphalt binder that can be directly related to binder performance.

[0008] Since asphalt is a thermoplastic material that softens as it is heated, selecting an asphalt binder the has a Performance Grade with a greater high pavement service temperature prevents, or at least reduces, pavement rutting due to traffic and general surface depression. This is particularly important in high temperature climates.

[0009] The use of rubbers in asphalt formulations tends to provide a better performing road with increased resilience to traffic and loads. Typical rubbers include fossil and/or virgin styrene butadiene styrene (SBS) rubbers and/or recycled ground tire rubber (GTR). GTR tends to be more cost effective. Additionally, the use of GTR has a positive environmental impact as it recycles waste tires that would otherwise end up in landfills. However, GTR is often not used due to its cross-linked nature which can increase asphalt viscosity and make the asphalt more difficult to process. In addition, the stability of GTR in asphalt can be poor, leading to separation or settling of the rubber.

[0010] Waxes can be employed to modify asphalt. One process is disclosed in International Application PCT/CA2017/050172 entitled “Polymer-Modified Asphalt with Wax Additive” which is hereby incorporated by reference. Waxes are compatible with a wide variety of asphalt additives and can be combined with a variety of materials commonly employed to improve the quality of asphalts.

[0011] Such waxes can be generated from plastic feedstocks including solid waste. A process to form synthetic waxes from solid waste is discussed in U.S. Patent No. 8,664,458 “Kumar”. U.S. Patent No. 8,664,458 and is hereby incorporated by reference.

[0012] A method of employing waxes produced from thermal degradation and/or catalytic depolymerization of plastic feedstocks to improve the physical properties of asphalt formulations, reduce emissions of VOCs in asphalt formulations, and/or improve the performance grade of asphalt binders such as paving asphalt and allow for greater incorporation and use of GTR would be commercially advantageous, environmentally responsible and a public health benefit. In some embodiments, these waxes could help adjust the resistance to flow and hardness of the asphalt independent of oxidation. The use of these waxes could reduce, if not eliminate, the need for oxidization.

Summary of the Invention

[0013] An asphalt formulation can include an asphalt blend and a wax made from polymeric material.

[0014] In some embodiments, the wax is made by catalytic depolymerization of the polymeric material. In other embodiments, the wax is made by thermal degradation of the polymeric material.

[0015] In certain embodiments, the polymeric material is polypropylene. In some embodiments, the polymeric material is polyethylene. In some embodiments, the polymeric material is polystyrene. In some embodiments, the polymeric material is a mixture of polyethylene, polypropylene, and/or polystyrene. In at least some embodiments, the polymeric material comprises recycled plastics. In some embodiments, the asphalt formulation can include additional modifiers such as ground tire rubber, SBS, and various polymers.

[0016] In certain embodiments, the wax is in the range of 0.5% to 25% by weight of the asphalt formulation. In certain embodiments, the wax is in the range of 3% to 5% by weight of the asphalt formulation. In certain embodiments, the wax is in the range of 0.5% to 3% by weight of the asphalt formulation. In certain embodiments, the wax is 5% by weight of the asphalt formulation.

[0017] In some embodiments, the wax is a low viscosity polyethylene or polypropylene wax. In other embodiments, the wax is a high viscosity polyethylene or polypropylene wax. Addition of the wax can increase the softening point of the asphalt, decrease the penetration depth of the asphalt and/or reduce, if not eliminate, the amount of time required for asphalt oxidation.

[0018] In some embodiments, the asphalt formulation can be made by addition a polyethylene wax to an asphalt blend.

[0019] In some embodiments, the wax is in the range of 0.5% to 10% by weight of the asphalt formulation.

[0020] Addition of the wax to the asphalt formulation can increase the high service temperature of the asphalt formulation alone or in the presence of other modifiers.

[0021] In certain embodiments, the asphalt formulation is a paving asphalt binder.

[0022] A method of improving the performance grade of a paving asphalt binder can include adding a polymeric wax to the asphalt binder alone or in the presence of other modifiers.

[0023] In some embodiments, a method of manufacturing an asphalt formulation can include adding a wax made from a polymeric material to an asphalt blend. In some embodiments, a method of manufacturing an asphalt formulation can include adding a polyethylene and/or polypropylene wax derived from polymeric feedstock to an asphalt blend.

[0024] In some embodiments, a method of improving the properties of an oxidized asphalt can include adding a polyethylene and/or polyethylene wax derived from polymeric feedstock to an oxidized asphalt.

[0025] In some embodiments, the asphalt formulation can have a first modifier, a second modifier and a wax, wherein the wax is made from a polymeric material. In some embodiments, the first modifier is ground tire rubber and the second modifier is a polymer.

[0026] In some embodiments, the wax is made by catalytic and/or thermal depolymerization of a polymeric material.

[0027] In some embodiments, a method of manufacturing an asphalt formulation can include adding a wax made from a polymeric material to an asphalt formulation.

[0028] In some embodiments, a method of improving binder performance grade of a paving asphalt binder includes adding a polypropylene wax to the asphalt binder. In some embodiments, a method of improving binder performance grade of a paving asphalt binder can include adding a polypropylene wax and at least one modifier to the asphalt binder, wherein the modifier has a different composition than the wax.

Brief Description of the Drawings

[0029] FIG. 1 is a bar graph illustrating the softening point of various asphalt formulations.

[0030] FIG. 2 is a bar graph illustrating the penetration depth of various asphalt formulations at 25°C.

[0031] FIG. 3 is a graph showing the true grade and performance grade high and low service temperatures of various asphalt formulations.

[0032] FIG. 4 is a graph showing the results of a Multiple-Stress Creep-Recovery (MSCR) of various asphalt formulations.

[0033] FIG. 5 is a graph showing the viscosity of various asphalt formulations at various temperatures.

Detailed Description of Illustrative Embodiment(s)

[0034] Various waxes generated from plastic feedstocks can be used to modify asphalt formulations. In some embodiments, the wax is made by catalytic depolymerization of polymeric material. In some embodiments, the wax is made by depolymerizing and/or thermally degrading polymeric material. In some embodiments, the catalyst used is a zeolite or alumina supported system or a combination of the two. In some embodiments, the catalyst is [Fe-Cu-Mo-P]/Al_2O_3.

[0035] In some embodiments, the catalyst is prepared by binding a ferrous-copper complex to an alumina or zeolite support and reacting it with an acid comprising metals and non-metals to obtain the catalyst material. In some embodiments, the catalyst comprises Al, Fe, Cu, and O, prepared by binding ferrous and copper complexes to an alumina and/or zeolite support. Other suitable catalyst materials include, but are not limited to, zeolite, mesoporous silica, H-mordenite and alumina.

[0036] In some embodiments, the wax is made by catalytically depolymerizing and/or thermally degrading polymeric material. In some embodiments, depolymerization can occur through the action of free radical initiators or the exposure to radiation.

[0037] In some embodiments, the polymeric material is polyethylene. In some embodiments, the polymeric material is polypropylene. In some embodiments, the polymeric material is polystyrene. The polymeric material can be polypropylene (PP), polystyrene (PS), high density polyethylene (HDPE), low density polyethylene (LDPE), linear low density polyethylene (LLDPE), and/or other variations of polyethylene.

[0038] In other embodiments, the polymeric material includes both polyethylene and polypropylene material. In some embodiments, the polymeric material is divided evenly by weight between polyethylene and polypropylene. In some embodiments, the polymeric material can contain up to 20% PP, lower levels of polystyrene, polyethylene terephthalate (PET), ethylene-vinyl acetate (EVA), (polyvinyl chloride) PVC, (ethylene vinyl alcohol) EVOH, and undesirable additives and/or contaminants, such as fillers, dyes, metals, various organic and inorganic additives, moisture, food waste, dirt, and/or other contaminating particles.

[0039] In other embodiments, the polymeric material includes combinations of LDPE, LLDPE, HDPE, and PP.

[0040] In some embodiments, the polymeric material comprises recycled plastics. In other or the same embodiments, the polymeric material comprises recycled plastics and/or virgin plastics.

[0041] In some embodiments, the polymeric material includes waste polymeric material feed. Suitable waste polymeric material feeds include mixed polystyrene waste, mixed polyethylene waste, mixed polypropylene waste, and/or a mixture including mixed polyethylene waste and/or mixed polypropylene waste. The mixed polyethylene waste can include LDPE, LLDPE, HDPE, PP, or a mixture including combinations of LDPE, LLDPE, HDPE, and/or PP. In some embodiments, the mixed polyethylene waste can include film bags, milk jugs or pouches, totes, pails, caps, agricultural film, and/or packaging material. In some embodiments, the mixed polypropylene waste can include carpet fibers, bottle caps, yogurt containers, bottle labels. In some embodiments, the mixed polystyrene waste can include food packaging containers, insulation, and electronic packaging. In some embodiments, the waste polymeric material feed includes up to 10% by weight of material other than polymeric material, based on the total weight of the waste polymeric material feed.

[0042] In some embodiments, the polymeric material is one of, or a combination of, virgin polyethylene (any one of, or combinations of, HDPE, LDPE, LLDPE and medium-density polyethylene (MDPE)), virgin polypropylene, or post-consumer, or post-industrial, polyethylene and/ or polypropylene (exemplary sources including bags, jugs, bottles, pails, and/or other items containing PE and/or PP).

[0043] In some embodiments, the addition of the wax changes the physical characteristics of the asphalt including:

- increasing the softening point of asphalt;
- decreasing the penetration of the asphalt;
- reducing the time required for asphalt oxidation
- lowering the formulation viscosity and/or
- increasing the stiffness of the asphalt.

[0044] In some embodiments, the percentage of wax in the asphalt formulation can be 0.5% to 25% by weight. In some preferred embodiments, the percentage of wax in the asphalt formulation can be 2% to 20% by weight. In some more preferred embodiments, the percentage of wax in the asphalt formulation can be 5% to 15% by weight. In some embodiments, the percentage of wax in the asphalt formulation can be 0.5% to 10% by weight.

[0045] In some embodiments, the asphalt formulation can include base asphalt, asphalt extender, asphalt flux, ground tire rubber, styrene-butadiene-styrene (SBS), cross linking agent, fillers, atactic polypropylene (APP), polypropylene, and polyethylene, Styrene Ethylene Butylene Styrene (SEBS), and/or Polyphosphoric Acid (PPA).

[0046] In at least some embodiments, the wax is incorporated into asphalt used in roofing asphalts, paving asphalts, asphalt emulsions, cut back asphalts, tack coats, crack fillers, adhesives and other products for waterproofing and joint sealing. In at least some embodiments, the wax can be incorporated into oxidized asphalt such as coating-grade asphalt and mopping-grade asphalt. In other embodiments, the wax can be incorporated into non-oxidized asphalt such as saturant-grade asphalt.

[0047] Oxidized asphalt can employ a variety of waxes, including those with melting points between and inclusive of 500-170 °C and viscosities between and inclusive of 10-25,000 cps. In some preferred embodiments, the wax(es) employed have melting points between and inclusive of 60-170 °C and viscosities between and inclusive of 10-10,000 cps. In some more preferred embodiments, the wax(es) employed have melting points between and inclusive of 110-170 °C and viscosities between and inclusive of 10-1000 cps.

[0048] Changes in melting point, viscosity, molecular weight, and/or polymer backbone structure of the wax can change the properties of the asphalt mixture. In general, addition of waxes will increase the softening point of the asphalt due to the polymers having higher softening points than the asphalt mixture. In general, addition of waxes will lower viscosities at formulating temperatures.

Example 1: Addition of Polyethylene Waxes to Mopping Asphalt

[0049] In a first example, effects of the addition of a wax formed via depolymerization of polyethylene were observed, As set forth in Tables 1-3, unmodified mopping asphalt served as a control.

Table 1: Sample Data Components

Ingredient	Grade/Type	Source
Asphalt Type IV	Mopping asphalt	Mid-States
HV Polyethylene Wax	AW115HV	GreenMantra
LV Polyethylene Wax	AW115LV	GreenMantra

Table 2: Asphalt Components as Percentage of Total Weight

	Formulation				
	Control I	A	B	C	D
Asphalt Type IV	100	97	95	97	95
AW115HV	0	3	5	0	0
AW155LV	0	0	0	3	5

Table 3: Asphalt Properties

		Formulation				
		Control I	A	B	C	D
Properties	Softening Point (°C)	103	117	124	114	123
	Penetration at 25°C (dmm)	15	11	11	12	12
	Viscosity at 75 cps (°C)	255	271	278	268	271
	Flashpoint by Cleveland Open Cup (°C)	280	274	266	276	268
	Ductility (cm)	3.18	2.86	1.91	2.54	1.91

[0050] Asphalt blends were prepared by mixing oxidized mopping asphalt with AW115HV or AW115LV wax at either 3% or 5% by weight. Mixing was performed by low shear mixers at elevated temperatures.

[0051] As set forth in Table 2, Control I Formulation consisted of 100% by weight of mopping asphalt.

[0052] Wax Blend Formulation A consisted of 97% by weight of mopping asphalt and 3% by weight of AW115HV.

[0053] Wax Blend Formulation B consisted of 95% by weight of mopping asphalt and 5% by weight of AW115HV.

[0054] Wax Blend Formulation C consisted of 97% by weight of mopping asphalt and 3% by weight of AW115LV.

[0055] Wax Blend Formulation D consisted of 95% by weight of mopping asphalt and 5% by weight of AW115LV.

[0056] The softening point of the formulations were determined using ASTM Method D36, the penetration of the formulations were determined using ASTM Method D5, the viscosity of the formulations were determined using ASTM Method D4402, the flashpoint of the formulations were determined using ASTM Method D92, and the ductility of the formulations were determined using ASTM Method D113.

[0057] The following conclusions can be drawn from the above test results: addition of 3% or 5% by weight of AW115LV wax increased the softening point and decreased the penetration depth of the oxidized mopping asphalt compared to Control I. Similarly, addition of 3% or 5% by weight of AW115HV wax increased the softening point of the oxidized mopping asphalt compared to Control I.

[0058] More specifically, the addition of AW115HV wax increased the softening point of the mopping asphalt by 12% or 18% and decreased the penetration depth of the mopping asphalt by 27%.

[0059] The addition of AW115LV wax increased the softening point of the mopping asphalt by 10% or 18% and decreased the penetration depth of the mopping asphalt by 20%.

[0060] Increasing the softening point and decreasing the penetration depth of oxidized mopping asphalt provides the following benefits:

- reducing the time required for asphalt oxidation;
- increasing the asphalt resistance to flow at high temperatures;
- improving the hardness properties of the asphalt;
- allowing for greater control of tailoring the physical properties of the asphalt; and
- handling variations in the supply stream.

[0061] Reduced asphalt oxidation time can be advantageous as it lowers production costs and/or emissions and allows material to be manufactured in a shorter amount of time.

[0062] FIG. 1 is a bar graph illustrating the softening point of various asphalt formulations. Softening points were measured according to ASTM D36 standards.

[0063] FIG. 2 is a bar graph illustrating the penetration depth of various asphalt formulations at 25°C. Penetration was measured according to ASTM D5 standards.

[0064] In some embodiments, the percentage of wax in the asphalt formulation, blend, or flux is 5% percent by weight. In some embodiments, the percentage of wax in the asphalt formulation, blend, or flux is between and inclusive of 0.5% to 15% percent by weight. In some embodiments, the percentage of wax in the asphalt formulation, blend, or flux is between and inclusive of 0.5% to 10% percent by weight.

[0065] In some embodiments, the asphalt formulation can include base asphalt, asphalt extender, asphalt flux, styrene-butadiene-styrene (SBS), cross linking agent and/or fillers.

[0066] In at least some embodiments, the wax is incorporated into an asphalt flux that can be used in roofing asphalts, paving asphalts, crack fillers, adhesives and/or other products for waterproofing and joint sealing. In at least some embodiments, the wax can be incorporated into oxidized asphalt such as coating-grade asphalt and mopping-grade asphalt. In other embodiments, the wax can be incorporated into non-oxidized asphalt such as saturant-grade asphalt.

[0067] Asphalt flux and various asphalt formulations can employ a variety of waxes, including those with melting points between and inclusive of 100-170°C and viscosities between and inclusive of 10-5000cps.

[0068] Changes to the wax, including but not limited to its molecular weight, and/or polymer backbone structure, can change the properties of the asphalt mixture.

[0069] Other potential benefits include increasing the shelf life of an asphalt formulation and extending the lifespan of roofing and coating materials that use a wax-modified asphalt formulation.

[0070] In some embodiments, waxes can be used in asphalt binders to increase performance grade. Such modifications can make the asphalt more stable at higher temperatures. Wax-modified asphalt binders can be used in applications such as patching, paving and coating.

[0071] In some embodiments, the addition of the wax improves the performance grade of an asphalt binder alone or in conjunction with other modifiers/additives by increasing the high service temperature. In certain embodiments, the modifiers can be ground tire rubber and various polymers. Increasing the high service temperature of asphalt provides the following benefits:

- increasing asphalt stability at higher temperatures, making it better suited for use in hot climates;
- preventing softening and deformation of pavement due to traffic; and/or lowering manufacturing costs.

[0072] In some embodiments the wax allows for GTR to be used with or as a replacement of SBS, offsetting it by 1-100%, without negatively affecting the asphalt formulation.

<u>Example 2: Addition of Polypropylene Wax to Asphalt Binder</u>

[0073] In at least some embodiments, the wax is incorporated into asphalt used in paving asphalts, crack fillers, adhesives and other products for waterproofing and joint sealing. In at least some embodiments, the wax can be incorporated into oxidized asphalt such as coating-grade asphalt and mopping-grade asphalt. In other embodiments, the wax can be incorporated into non-oxidized asphalt such as saturant-grade asphalt.

[0074] In some embodiments, waxes can be used to modify paving asphalt binder. Paving asphalt binder can employ a variety of waxes, including those with melt points between and inclusive of 60-170° Celsius, and viscosities between and inclusive of 5-3000cps. In some preferred embodiments, the wax(es) employed have melt points between and inclusive of 110-170°C and/or viscosities between, and including, 15-1000 cps.

[0075] In some preferred embodiments, a polypropylene wax can be used to improve performance grade of paving asphalt binder.

[0076] Changes in melting point, viscosity, molecular weight, and/or polymer backbone structure of the wax can change the properties of the asphalt mixture.

Table 4: Sample Data Components

Ingredient	Grade/Type	Source
Asphalt Binder	Paving Grade Asphalt (PG58-28)	Commercial Stock
Asphalt Modifier	Ground Tire Rubber	Commercial Stock
Asphalt Modifier	Polymer	Commercial Stock
Polypropylene Wax	A155 wax	GreenMantra (Applicant)

Table 5: Asphalt Components as Percentage of Total Weight

	Formulation				
	Control II	E	F	G	H
PG58-28	100	99.5	97	86.5	84
Ground Tire Rubber	0	0	0	10	10
Polymer	0	0	0	3	3
A155 wax	0	0.5	3	0.5	3

[0077] Asphalt formulations were prepared by mixing asphalt binder with various modifiers including ground tire rubber (GTR), polymer, and/or A155 wax.

[0078] As set forth in Table 5, Control II consisted of unmodified asphalt binder.

[0079] Asphalt Formulation E consisted of 99.5% by weight of asphalt binder and 0.5% by weight of A155 wax.

[0080] Asphalt Formulation F consisted of 97% by weight of asphalt binder and 3% by weight of A155 wax.

[0081] Asphalt Formulation G consisted of 86.5% by weight of asphalt binder, 10% by weight of GTR, 3% by weight of polymer and 0.5% by weight of A155 wax.

[0082] Asphalt Formulation H consisted of 84% by weight of asphalt binder, 10% by weight of GTR, 3% by weight of polymer and 3% by weight of A155 wax.

[0083] Binder testing to measure true grade of the paving asphalt formulations included the Rotational Viscometer Test, the Dynamic Shear Rheometer test, the Bending Beam Rheometer test and the Direct Tension Test.

Table 6: True Grade and Performance Grade of Formulations

	Formulation									
	Control II		E		F		G		H	
	High	Low	High	Low	High	Low	High	Low	High	Low
True Grade (°C)	60.8	-28.7	61.6	-28.2	66.5	-20	82.6	-26.3	91.5	-25.8
Performance Grade (°C)	58	-28	58	-28	64	-16	82	- 22	88	-22

[0084] As can be seen above, the addition of A155 wax improved the performance of the asphalt binder at high temperatures alone and in conjunction with other modifiers including GTR and polymer. This change can be seen by comparing the Performance Grade high service temperature of each formulation with the upper temperature reported from binder testing of each formulation (reported as true grade values).

[0085] The ability of A155 wax to improve the high service temperature of asphalt binder in the presence of GTR and polymer indicates that the A155 wax facilitates incorporation of asphalt modifiers. This is advantageous because it can lower the production cost associated with mixing GTR into paving asphalt, a process that requires greater high shear mixing when compared to SBS for incorporation into asphalt.

[0086] Alternatively, production cost could be lowered by adding the A155 wax and a lower amount of GTR to the asphalt binder without compromising the performance grade of the final paving asphalt product. This can produce an asphalt formulation with a much lower viscosity that flows faster and is easier to process.

[0087] Turning first to the results for Control II, unmodified asphalt binder had a true grade temperatures of 60.8°C and -28.7°C. Addition of A155 wax at 0.5% by weight (Formulation E) or 3% by weight (Formulation F) to the asphalt binder increased the true grade high service temperature by 0.8°C and 5.7°C, respectively, compared to the true grade high service temperature of Control II. 0.5% of A155 wax increased the high service temperature to 61.6°C which is 3.6 degrees greater than the performance grade high service temperature of 58°C for the same formulation. 3% of A155 wax increased the high service temperature to 66.5°C which is 2.5 degrees greater than the performance grade high service temperature of 64°C for the same formulation. These data demonstrate that the A155 wax directly affects and improves asphalt binder performance at high temperatures.

[0088] Comparing Control II with the addition of both the wax and the GTR led to even greater results. Addition of GTR at 10% by weight, polymer at 3% by weight, and A155 wax at 0.5% by weight (Formulation G) or GTR at 10% by weight, polymer at 3% by weight, and A155 wax at 3% by weight (Formulation H) increased the true grade high service temperature by 21.8°C and 30.7°C, respectively, compared to the true grade high service temperature of Control II. 0.5% of A155 wax increased the high service temperature to 82.6°C which is 0.6 degrees greater than the performance grade high service temperature of 82°C for the same formulation. 3% of A155 wax increased the high service temperature to 91.5°C which is 3.5 degrees greater than the performance grade high service temperature of 88°C for the same formulation. These data points demonstrate that the A155 wax enhances the ability of other asphalt modifiers, GTR and a polymer in this instance, to improve asphalt binder performance at high temperatures.

[0089] FIG. 3 is a graph showing the true grade and performance grade high and low service temperatures of various asphalt formulations.

[0090] Increasing the high service temperature of asphalt, as well as addition of GTR, can provide at least one, if not all, of the following benefits:

- increasing asphalt stability at higher temperatures, making it better suited for use in hot climates;
- preventing softening and deformation of pavement due to traffic;
- increasing road elasticity or recovery under various weather and/or load-related stresses;
- lowering the formulation cost compared to use of SBS; and/or
- reducing the amount of GTR in landfills.

Example 3: Addition of Wax to Asphalt Binder

Table 7: Sample Data Components

Ingredient	Grade/Type	Source
Asphalt Binder	Paving Grade Asphalt (PG64-22)	Commercial Stock
Polyethylene Wax	A115 wax	GreenMantra (Applicant)
Polyethylene Wax	A120 wax	GreenMantra (Applicant)
Polyethylene Wax	A125 wax	GreenMantra (Applicant)
Polypropylene Wax	A155 wax	GreenMantra (Applicant)

Table 8: Asphalt Components as Percentage of Total Weight

	Formulation				
	Control III	I	J	K	L
PG64-22	100	97	97	97	97
A115 wax	0	3	0	0	0
A120 wax	0	0	3	0	0
A125 wax	0	0	0	3	0
A155 wax	0	0	0	0	3

[0091] Asphalt formulations were prepared by mixing asphalt binder with the various waxes.

[0092] As set forth in Table 8, Control II consisted of an unmodified asphalt binder.

[0093] Asphalt Formulation I consisted of 97% by weight of asphalt binder and 3% by weight of A115 wax.

[0094] Asphalt Formulation J consisted of 97% by weight of asphalt binder and 3% by weight of A120 wax.

[0095] Asphalt Formulation K consisted of 97% by weight of asphalt binder and 3% by weight of A125 wax.

[0096] Asphalt Formulation L consisted of 97% by weight of asphalt binder and 3% by weight of A155 wax.

[0097] Binder testing to measure true grade of the paving asphalt formulations included the Rotational Viscometer Test, the Dynamic Shear Rheometer test, the Bending Beam Rheometer test and the Direct Tension Test.

Table 9: True Grade and Performance Grade of Formulations

	Formulation									
	Control III		I		J		K		L	
	High	Low	High	Low	High	Low	High	Low	High	Low
True Grade (°C)	69.3	-24.0	74.6	-21.1	72.2	-20.1	75.6	-21.5	79.2	-22.7
Performance Grade (°C)	64	-22	70	-16	70	-16	70	-16	76	-22

[0098] As can be seen above, the addition of waxes improved the performance of the asphalt binder at high temperatures. This change can be seen by comparing the Performance Grade high service temperature of each formulation with the upper temperature reported from binder testing of each formulation (reported as true grade values).

[0099] The addition of the polypropylene wax (Formulation L) had the greatest effect on the Performance Grade high service temperature. This is due to the higher softening point of the wax which in turn increases the softening point of the asphalt, leading to a stiffer asphalt at the temperatures included in the testing.

[00100] Increasing the high service temperature of asphalt can provide at least one, if not all, of the following benefits:

- increasing asphalt stability at higher temperatures, making it better suited for use in hot climates;
- preventing softening and deformation of pavement due to traffic;
- increasing road elasticity or recovery under various weather and/or load-related stresses; and/or
- lowering the formulation cost compared to use of SBS.

[00101] The ability of the waxes to improve the high service temperature of asphalt binder waxes facilitate incorporation of asphalt modifiers.

Table 10: Multiple-Stress Creep-Recovery of Formulations

	Formulation				
	Control III	I	J	K	L
0.1- Rolling Thin-Film Oven	1.650	0.600	1.100	0.565	0.175
3.2- Rolling Thin-Film Oven	1.750	1.000	1.300	0.860	0.645

[00102] Non-recoverable creep compliances of the formulations were measured at two different stress levels (.1 and 3.2). Results are shown in kPa^{-1}. In determining traffic rating, AASHTO M332 uses the 3.2 stress level, and the cutoffs are ≤2.0 for heavy traffic (H) and ≤1.0 for Very Heavy traffic (V). In this case both K and L showed noted benefits.

[00103] FIG. 4 is a graph showing the results of a Multiple-Stress Creep-Recovery (MSCR) of various asphalt formulations.

[00104] The MSCR was conducted according to AASHTO M332. As seen in Table 10 and FIG. 4, an improvement in non-recoverable creep compliance (J_{nr}) was seen in each Formulation incorporating a wax. This indicates an increase in rut resistance and the ability to handle heavier traffic loads. For Formulation L, an improvement of the traffic designation of the MSCR based performance grade with a possible increase from 64H-22 to 64V-22 was observed.

Example 4 – Displacement of SBS in SBS Modified Asphalt

Table 11: Sample Data Components

Ingredient	Grade/Type	Source
Asphalt Binder	Paving Grade Asphalt (PG64-22)	Commercial Stock
Asphalt Modifier	SBS	Commercial Stock
Polyethylene Wax	A115 wax	GreenMantra (Applicant)
Polypropylene Wax	A155 wax	GreenMantra (Applicant)

Table 12: Asphalt Components as Percentage of Total Weight

	Formulation		
	Control IV	M	N
PG64-22	97	97	97
SBS	3	2	2
A115 wax	0	0	1
A155 wax	0	1	0

Table 13: True Grade and Performance Grade of Formulations

	Formulation					
	Control IV		M		N	
	High	Low	High	Low	High	Low
True Grade (°C)	79.9	-8.3	79.9	-11.0	78.7	-6.6

[00105] Table 13 shows an improvement of the true grade in the high temperature range, modifying a binder containing SBS by offsetting with waxes. This shows an improvement in decreasing deformation, and increased stability at high temperatures.

Table 14: Viscosity Measurements of Formulations

	Formulation		
	Control IV	M	N
cP at 135°C	6168.75	Still semisolid	4312.5
cP at 165°C	1668.75	1175	1225

[00106] FIG. 5 is a graph showing the viscosity of various asphalt formulations at various temperatures. FIG. 5 illustrates the lowering of the viscosity of an SBS modified binder by offsetting SBS with waxes. This reduction in viscosity indicates the ability to facilitate incorporation of other modifiers.

[00107] Binder testing followed AASHTO M320 and M322 for performance grade and included rotational viscosity testing, dynamic shear rheometry, bending beam rheometry, and aging methods including a rolling thin film oven test, and pressure aging vessel. The true grade was determined from the data obtained by these tests.

[00108] While particular elements, embodiments and applications of the present invention have been shown and described, it will be understood, that the invention is not limited thereto since modifications can be made without departing from the scope of the present disclosure, particularly in light of the foregoing teachings.

CLAIMS

What is claimed is:

1. An asphalt formulation comprising an asphalt blend and a wax, wherein said wax is made from a polymeric material.

2. The asphalt formulation of claim 1 wherein said wax is made by catalytic depolymerization of said polymeric material.

3. The asphalt formulation of claim 1 wherein said wax is made by thermal degradation of said polymeric material.

4. The asphalt formulation of claim 1 wherein said polymeric material is polypropylene.

5. The asphalt formulation of claim 1 wherein said polymeric material is polyethylene.

6. The asphalt formulation of claim 1 wherein said polymeric material is a combination of polyethylene, polypropylene and/or polystyrene.

7. The asphalt formulation of claim 1 wherein said polymeric material comprises recycled plastics.

8. The asphalt formulation of claim 1 wherein said wax is in the range of 0.5% to 25% by weight of said asphalt formulation.

9. The asphalt formulation of claim 8 wherein said wax is a low viscosity polyethylene or polypropylene wax derived from polymeric feedstock.

10. The asphalt formulation of claim 8 wherein said wax increases the softening point of said asphalt formulation.

11. The asphalt formulation of claim 8 wherein said wax decreases the penetration depth of said asphalt formulation.

12. The asphalt formulation of claim 8 wherein said wax reduces the amount of time required for asphalt oxidation.

13. The asphalt formulation of claim 8 wherein said wax is a high viscosity polyethylene or polypropylene wax derived from polymeric feedstock.

14. The asphalt formulation of claim 13 wherein said wax increases the softening point of said asphalt formulation.

15. The asphalt formulation of claim 13 wherein said wax decreases the penetration depth of said asphalt formulation.

16. The asphalt formulation of claim 13 wherein said wax reduces the amount of time required for asphalt oxidation.

17. A method of manufacturing an asphalt formulation comprising adding a wax made from a polymeric material to an asphalt blend.

18. A method of manufacturing an asphalt formulation comprising adding a polyethylene or polypropylene wax derived from polymeric feedstock to an asphalt blend.

19. A method of improving the properties of an oxidized asphalt comprising adding a polyethylene or polyethylene wax derived from polymeric feedstock to said oxidized asphalt.

20. The asphalt formulation of claim 1 wherein said polymeric material is polystyrene.

21. The asphalt formulation of claim 18 wherein said wax is in the range of 0.5% to 25% by weight of said asphalt formulation.

22. The asphalt formulation of claim 1 wherein said asphalt formulation is a paving asphalt binder.

23. The asphalt formulation of claim 1 wherein said wax increases a high service temperature of said asphalt formulation.

24. An asphalt formulation comprising an asphalt formulation a first modifier, a second modifier and a wax, wherein said wax is made from a polymeric material.

25. The asphalt formulation of claim 24 wherein said wax is made by catalytic depolymerization of said polymeric material.

26. The asphalt formulation of claim 24 wherein said wax is made by thermal degradation of said polymeric material.

27. The asphalt formulation of claim 24 wherein said polymeric material is polypropylene.

28. The asphalt formulation of claim 24 wherein said polymeric material is polyethylene.

29. The asphalt formulation of claim 24 wherein said first modifier is ground tire rubber and said second modifier is a polymer.

30. The asphalt formulation of claim 24 wherein said wax is in the range of 0.5% to 25% by weight of said asphalt formulation.

31. The asphalt formulation of claim 24 wherein said asphalt formulation is a paving asphalt binder.

32. The asphalt formulation of claim 24 wherein said wax increases a high service temperature of said asphalt formulation.

33. A method of manufacturing an asphalt formulation comprising adding a wax made from a polymeric material to an asphalt formulation.

34. A method of manufacturing an asphalt formulation comprising adding a polypropylene wax, a first modifier and a second modifier to an asphalt formulation.

35. A method of improving binder performance grade of a paving asphalt binder comprising adding a polypropylene wax to said asphalt binder.

36. A method of improving binder performance grade of a paving asphalt binder comprising adding a polypropylene wax and at least one modifier to said asphalt binder, wherein said modifier has a different composition than said wax.

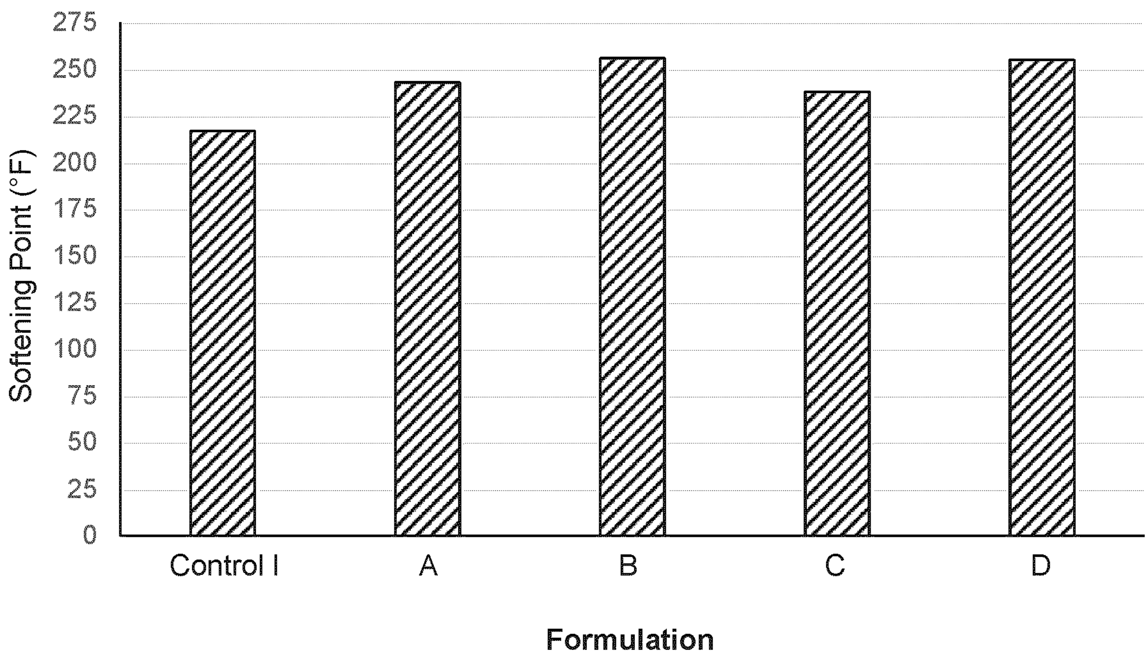
275
250
225
200
175
150
125
100
75
50
25
0
Softening Point (°F)
Control I
A
B
C
D
Formulation

FIG. 1

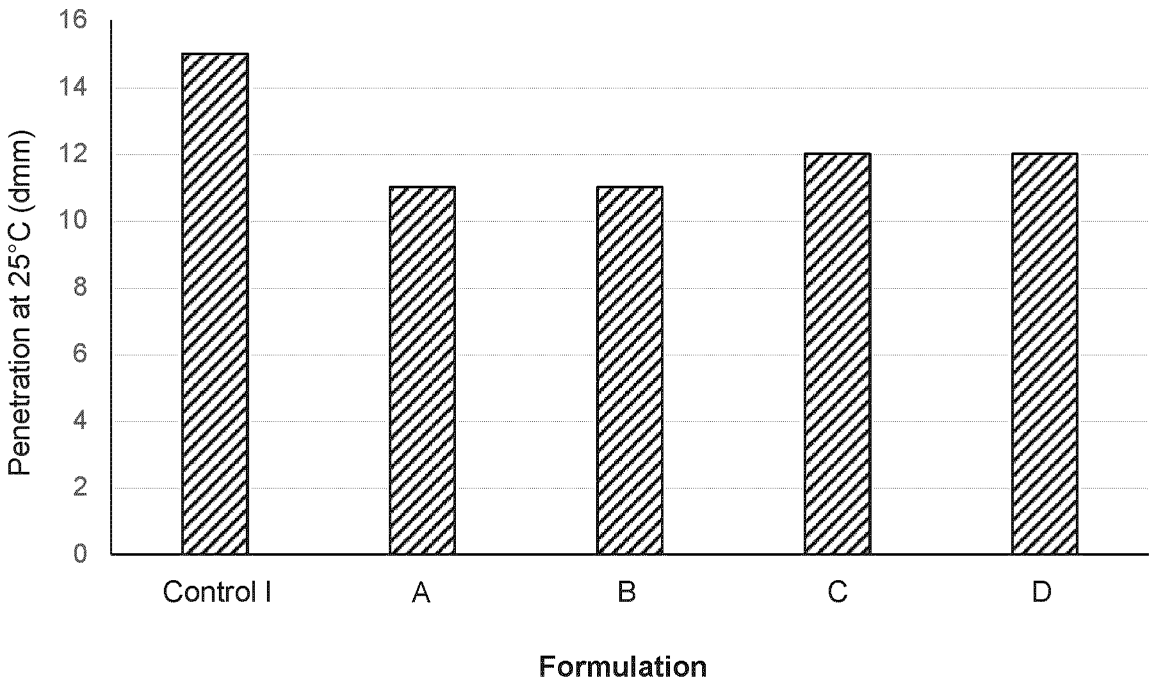
16
14
12
10
8
6
4
2
0
Penetration at 25°C (dmm)
Control I
A
B
C
D
Formulation

FIG. 2

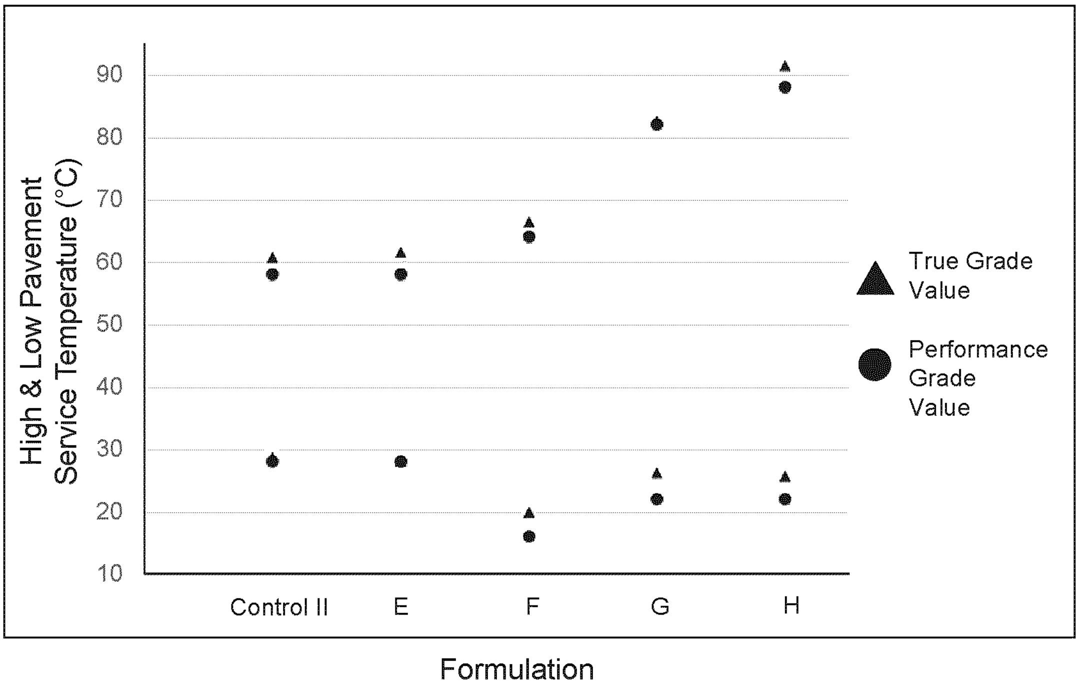
High & Low Pavement Service Temperature (°C)
90
80
70
60
50
40
30
20
10
Control II
E
F
G
H
True Grade Value
Performance Grade Value
Formulation

FIG. 3

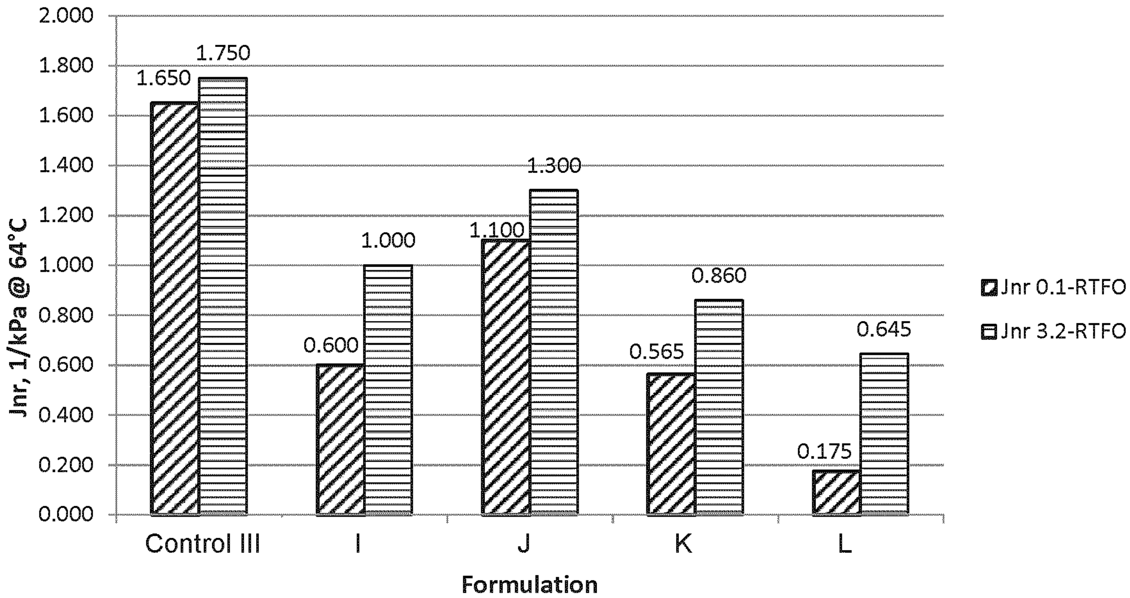
2.000
1.800
1.600
1.400
1.200
1.000
0.800
0.600
0.400
0.200
0.000
Jnr, 1/kPa @ 64°C
1.650
1.750
0.600
1.000
1.100
1.300
0.565
0.860
0.175
0.645
Control III
I
J
K
L
Formulation
Jnr 0.1-RTFO
Jnr 3.2-RTFO

FIG. 4

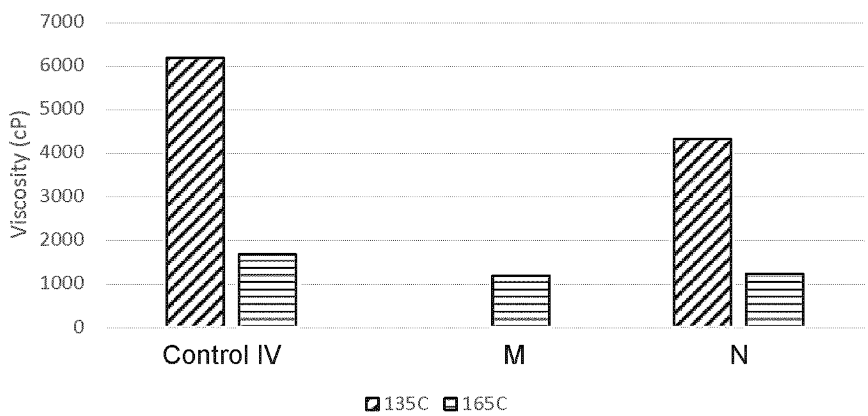
7000
6000
5000
4000
3000
2000
1000
0
Viscosity (cP)
Control IV
M
N
135C
165C

FIG. 5

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2019/050762

A. CLASSIFICATION OF SUBJECT MATTER
IPC: ***C08L 95/00*** (2006.01) , ***C04B 24/26*** (2006.01) , ***C04B 26/26*** (2006.01) , ***C08J 3/20*** (2006.01) , ***C08L 23/00*** (2006.01) , ***C08L 25/06*** (2006.01) ***C08L 91/06*** (2006.01)

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)
IPC: ***C08L 95/00*** (2006.01) , ***C04B 24/26*** (2006.01) , ***C04B 26/26*** (2006.01) , ***C08J 3/20*** (2006.01) , ***C08L 23/00*** (2006.01) , ***C08L 25/06*** (2006.01) , ***C08L 91/06*** (2006.01)

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

Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used)
Questel (Fampat), CIPO Library Discovery Tool (sample search terms: asphalt, wax, paving, polymer wax, polypropylene wax, polyethylene wax, polystyrene wax, polypropylene, polyethylene, polystyrene, depolymerized, ground tire rubber, and related terms)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2017/136957 A1 [DIMONDO, D. et al] 17 August 2017 (17-09-2017) (see entire document, especially paragraphs [0005], [0007], [0035] – [0039] and [0056] and Examples).	1-18, 21, 24-28, 30, 33 and 34
X	US 2010/0227954 A1 [NAIDOO, P. et al.] 09 September 2010 (09-09-2010) (see paragraphs [0024], [0027], [0029] – [0033], [0049] and [0062] –[0067]and Examples).	1, 3-6, 8, 10-17, 21-24 and 26-33
X	US 2014/0299017 A1 [PARVEZ, M. A. et al.] 09 October 2014 (09-10-2014) (see paragraphs [0005], [0016]-[0019], [0027], [0035]-[0037],[0042] and [0057] and Examples).	1,5 8, 10, 11, 17, 21-23 and 33
X	GERGÓ, P. et al. "Rheological Investigation of Rubber Bitumen Containing Various Waxes as Warm Mix Additive" STUDIA UBB CHEMIA LXII, 2 Tom II, **2017** page 247-257 (see entire document).	1, 3, 4, 8-13, 16-19, 21-23, 33, 35 and 36

☑ 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
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"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
08 July 2019 (08-07-2019)

Date of mailing of the international search report
17 July 2019 (17-07-2019)

Name and mailing address of the ISA/CA
Canadian Intellectual Property Office
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50 Victoria Street
Gatineau, Quebec K1A 0C9
Facsimile No.: 819-953-2476

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Owen Terreau (819) 639-9384

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2012/007833 A2 [KUMAR, A. et al.] 19 January 2012 (19-01-2012) (see entire document)	1-36

Patent Document Cited in Search Report	Publication Date	Patent Family Member(s)	Publication Date
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		BR112018016499A2	26 December 2018 (26-12-2018)
		CA3013953A1	17 August 2017 (17-08-2017)
		CN108779398A	09 November 2018 (09-11-2018)
		EP3414302A1	19 December 2018 (19-12-2018)
		JP2019507812A	22 March 2019 (22-03-2019)
		MX2018009808A	21 January 2019 (21-01-2019)
		US2018346683A1	06 December 2018 (06-12-2018)
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		US2015105494A1	16 April 2015 (16-04-2015)
		US2015105496A1	16 April 2015 (16-04-2015)
		US2015112001A1	23 April 2015 (23-04-2015)
		US2018312694A1	01 November 2018 (01-11-2018)
		US2015105495A1	16 April 2015 (16-04-2015)
		US2018155548A1	07 June 2018 (07-06-2018)
		US2019169437A1	06 June 2019 (06-06-2019)
US2014299017A1	09 October 2014 (09-10-2014)	CA2902610A1	16 October 2014 (16-10-2014)
		CN105143326A	09 December 2015 (09-12-2015)
		CN105143326B	21 July 2017 (21-07-2017)
		WO2014168860A1	16 October 2014 (16-10-2014)
WO2012007833A2	19 January 2012 (19-01-2012)	CA2805570A1	19 January 2012 (19-01-2012)
		CN103168016A	19 June 2013 (19-06-2013)
		CN104877699A	02 September 2015 (02-09-2015)
		EP2407528A1	18 January 2012 (18-01-2012)
		ES2516743T3	31 October 2014 (31-10-2014)
		JP2013539476A	24 October 2013 (24-10-2013)
		US2012016169A1	19 January 2012 (19-01-2012)