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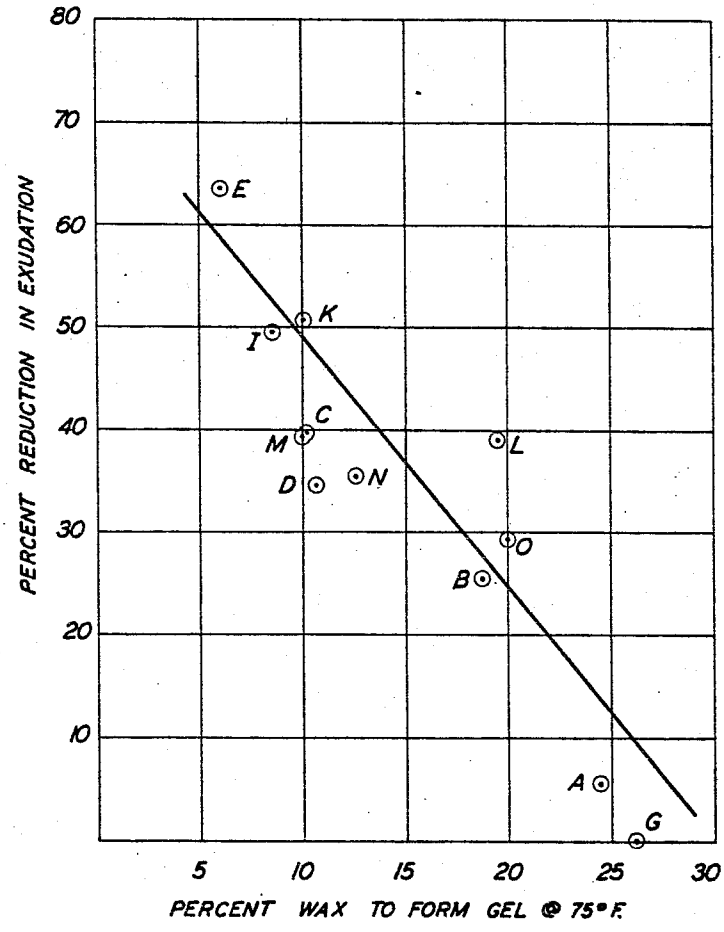
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WOOD PRESERVATION COMPOSITION AND METHOD

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OIL ADSORPTION CHARACTERISTICS VRS. PERFORMANCE IN REDUCING EXUDATION



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WOOD PRESERVATION COMPOSITION AND METHOD

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The present invention relates to the preservation of wood and protecting it from attack by various organisms including insects, plant and marine organisms. More particularly, this invention provides a mixture readily incorporated into wood by conventional commercial treating practices which will impart the desired wood preservative properties to wood and at the same time serve to decrease the rate and amount of loss of preservative composition from wood after completion of the treating process.

This application is a continuation-in-part of our application Serial No. 838,052 entitled: Wood Preservative Composition to Stabilize Treatment Within Wood Treated by a Pressure or Vacuum System, filed on September 4, 1959, and now abandoned.

The art of wood preservation has long practiced the forcing of various compositions into wood to impart preservative value to the treated wood. This protection aimed either to poison the wood so that fungi or insects would not consume the wood, or to add materials that would fill the cell voids with a water excluding or water repelling material, thus reducing the moisture held by the fibers to a point below the moisture requirements for fungus growth. In the former category would fall such products as the oil preservatives (creosote, creosote coal-tar, etc.), the oil-borne preservatives (pentachlorophenol, copper naphthenate, etc.) and the water-borne preservatives (consisting of combinations containing copper, chromium, fluorides, zinc, etc.). For filling the cell voids to produce water repellency, waxes such as montan wax have been used.

One of the problems that has plagued the wood preserving plant as well as the user of pressure treated wood poles, is what is known as bleeding or exudation of the preservative solution from the surfaces of the wood after treatment. In some cases this bleeding may take place soon after treatment; in other cases it may occur some time later when the treated wood is in storage yards or when the wood is placed in service.

Such losses are undesirable not only because of the problems involved in handling the wood which is wet with exudate, but also because of the loss of preservative which means that it is no longer in the wood to exert its preservative effect.

Attempts have been made by various methods to prevent this bleeding. One practice known to the wood preservation industry is to steam the wood after treatment, thus heating and expanding the treating solution in the outer cells of the wood so that it bleeds while the wood is still in the treating vessel. Such bleeding removes some of the treating solution from the outer wood cells and if solution from deeper in the wood later moves outward, it enters the unfilled surface and near-surface cells rather than oozing (bleeding) out of the wood.

Another method that has been employed is to use blocking agents which are in solution when applied to the wood, but which solidify later to block the movement of liquid preservative solution from the surface cells. Also, wood has been impregnated with high melting point waxes to produce water repellency. Coolidge disclosed the use of montan wax for such purposes in the 1926 Proceedings of the American Wood-Preservers' Association, pages

183 to 191, and United States Patents 1,556,570; 1,609,642; 1,648,294; and 1,648,295.

In these processes, increased costs are involved due to the necessity of keeping the treating solution at relatively high temperatures at all times, thus requiring steam jacketed or otherwise heated storage tanks, pipe lines and the like. Furthermore, large volumes of blocking agents are required with resultant increased costs of materials as well as appreciable increased weight of the treated wood items even after some of the carrier has been removed.

It is an object of the present invention to provide a wood treating composition that will be retained within the wood after application with a minimum of loss by bleeding.

It is a further object of the invention to provide a liquid wood treating composition that can be applied without expensive heating equipment necessary for maintaining normally-solid materials in a liquid state.

It is a further object of the invention to provide a wood treating composition that can be applied at reduced cos and will be retained within the wood with minimum loss by bleeding.

A further object of the invention is to provide a process for treating wood that is efficient and economical and will provide extended life of the treated wood.

It is a further object of the invention to provide a wood treating composition that holds a toxicant in its distributed positions within the wood because of reduced movement of the whole wood preservative composition within the wood.

A further object of the invention is to provide a wood treating composition wherein the concentration of toxicant in a given treated area within the wood is increased because the solvent leaves the wood by volatilization rather than by movement of the whole solution.

A further object of the invention is to provide a wood treating composition including a wax which holds a toxicant distributed deeply within the treated wood.

The present invention resides in the concept of a wood treating mixture including a preservative and wax or mixture of waxes which, when used as a treating solution, have the capacity to form a crystalline lattice with intercellular crystal spaces or apertures between crystals thereof that will permit a large quantity of the preservative to be held to the wax crystals and within the inter-crystalline spaces. Thereby, the mixture forms a gel after impregnation into the wood, said gel serving to fix the preservative substantially within the wood and reduce exudation of the preservative from the wood. The preservative used can be a single chemical toxicant or a mixture of toxicants with or without a carrier depending upon the nature of the preservative.

The mixture or composition of the invention provides for wood preservation at various temperatures and pressures. At relatively low temperatures, when the wax is solid, the above-described lattice fixes the carrier and toxicant within the wood and prevents exudation. At higher temperatures, such as those achieved when treated poles are exposed to sunlight, the waxes employed according to the invention reduce exudation and loss of the toxicant because of their molecular size and configuration which influence viscosity. At these elevated temperatures when there exists substantially a partial solution within the wood, the viscosity and molecular size and configuration of the wax tends to prevent the flow of the carrier and toxicant toward the surface of the wood.

To explain further, as the low boiling fractions of the preservative hydrocarbon carrier volatilize, the non-volatile toxicant and wax increase in concentration. As the concentration of the wax at the surface of the wood increases substantially the temperature at which the cloud

point and crystallization point occur is significantly raised. In the static system there also seems to be a retentive capacity of the wax molecule for the oil even though the lattice has not formed through crystallization of wax from solution. This is substantiated by the results obtained by various waxes in reducing exudation of preservative from wood (column 14 in table). As described below, the tests reported in the table were conducted and the test blocks maintained at a temperature (180° F.) well above the solution temperature of the whole original solution. It is also noted that the cloud point of the preferred combination exists over a broad range of temperatures and this contributes to blocking of the pit membranes and other apertures which prevent oil movement through the wood structure.

At temperatures where essentially the solid state exists, the wax employed in the mixture of the invention acts similar to a sponge to hold the carrier oil and toxicant within its crystalline lattice. By thus holding the carrier and toxicant in the lattice, the motility of the carrier and toxicant is reduced and the overall mixture assumes the nature of a gel. The wax must have a desired spacing between crystals thereof. If the crystals are too small, as in certain microcrystalline waxes, there is insufficient room between the crystals to accommodate and hold sufficient quantities of the carrier and toxicant to give the desired effect. On the other hand, if the wax crystals are too large, there is less surface area of crystal per unit volume for adsorbing the carrier oil, and the "holding power" of the wax for oils is insufficient.

Molecular configuration is another important factor in selecting a suitable wax for the mixture of the invention. Straight chain or normal paraffin waxes are undesirable per se because they form large crystals with excessively large openings therebetween. The surface tension of the carrier oil may be and generally is insufficient to hold the carrier oil and toxicant in these relatively large inter-crystalline spacings. The selection of the wax thus depends upon more than one characteristic of the wax. Among the important characteristics are the molecular weight, the configuration of the molecule and the crystalline lattice size in which it can be crystallized. It will be apparent from the above discussion that the size of the spacings between the crystals is important. It has been shown that there is an optimum range of spacing size between upper and lower ranges which is suitable for reducing the loss of the oil carrier and toxicant from treated wood. The numerical size of the desirable spacings between crystals is not yet known nor is a method known of making such measurement. However, according to the invention, numerous suitable waxes have been found which produce the desired results. Certain properties of these waxes have been measured and desirable ranges established so as to define the limits of the invention.

Among the physical properties of the waxes suitable for the composition and method of the invention are the following with the desired ranges:

Average molecular weight	350 to 600
A preferred molecular weight range within the foregoing broad range is a 450 to 500.	
Viscosity, Saybolt Universal seconds (SSU) @ 210° F.—seconds	40–80
A preferred range is 45–55 Saybolt Universal seconds.	
Refractive index n_d^{100} C	1.4230–1.4415
A preferred range is 1.4275–1.4325.	
Melting point range (ASTM: D87)—° F.	135 to 185
A preferred range is 155° F. to 165° F.	

While certain microcrystalline waxes fall within the range of this invention, a preferred wax would be defined as a distillate intermediate paraffin wax. A wax so described and falling within the preferred range of physical characteristics as indicated above would be one which, when combined with the toxicant and petroleum carrier, would penetrate the wood structure and not be filtered from solution to any great extent under the conditions of commercial application. Waxes of higher molecular weight (e.g. microcrystalline waxes) will in general be filtered from solution, thus changing the distribution characteristics of the preservative solution.

A desirable wax for the mixture of the invention would be one which would produce a very viscous solution throughout a normal range of temperatures roughly up to 120° F.; but which when heated to temperatures used in wood treating applications, would thin out sufficiently to permit penetration of the wood.

The molecular size and configuration of the wax are also important factors in selecting a wax for the invention. Such a wax should have a substantial quantity of non-normal or branch-chained paraffins such as iso-paraffins, monocycloalkanes as well as other cyclic hydrocarbons.

Seven examples of waxes which can be used as illustrative but which are not to be construed as limiting the scope of this patent, and which have imparted the desired characteristics to a preservative are petroleum hydrocarbon waxes of the microcrystalline residual and intermediate paraffin distillate waxes designated C, D, E, F, H, I, and K in the table below.

While pure or substantially pure waxes can be employed, less-refined or crude waxes can also be used which contain various amounts of residual oils. Further, blends of waxes can be employed to provide wax mixtures having properties as set forth above. For example, paraffin wax generally has a crystal structure with large crystals and large inter-crystal spaces. On the other hand, microcrystalline petroleum wax has a crystal structure with small crystals and small inter-crystal spaces. By blending relatively small quantities of certain microcrystalline petroleum wax with relatively large quantities of certain normal paraffin wax, a blend can be obtained having crystal structure suitable for use in the wood treating composition of the invention.

The wax is desirably employed in amounts generally not greater than 10% by weight of the entire mixture. Particularly advantageous results are obtained using 1% to 2% by weight of the preferred wax types.

Among the toxicants which can be employed are: oil-borne preservatives such as pentachlorophenol and copper naphthenate; oil preservatives such as creosote, creosote coal-tar and the like; et cetera.

Among the carrier or solvents which can be employed are petroleum oil solvents such as heavy and light solvent petroleums designated as AWPA P9 oils (American Wood-Preservers' Association), and equivalent oil solvents or carriers.

The treatment method according to the invention includes applying the mixture to wood by impregnating the wood with the mixture. Conventional pressure, thermal and vacuum processes can be employed. The following is a specific example of the mixture and method according to the invention.

Mix in a kettle the following three ingredients:

(1) 835 pounds of AWPA P9 light recycle oil having a density of 7.55 pounds per gallon, a viscosity measured at 100° F. of 37 Saybolt Universal seconds, a distillation range including initial boiling point of 474° F., 50% distilled over at 539° F., 90% distilled over at 620° F., and an end point of 668° F., a pour point of −15° F., a flash point of 209° F. by Pensky-Martens closed cup method and capable of dissolving in excess of 10% by weight of pentachlorophenol at 75° F.

(2) 45.5 pounds of technical grade pentachlorophenol, and

(3) 8.9 pounds of distillate intermediate paraffin wax melting at 159.6° F. as determined by American Society of Testing Materials, bulletin D–87, having a refractive index n_d^{100} of 1.4305, a viscosity measured at 210° F. of 51.4 Saybolt Universal seconds and designated as wax E in the table below.

Agitate the mixture for approximately 30 to 45 minutes while maintaining the temperature at about 160° F. until a clear solution is obtained.

As an example of employing the mixture in treating one specie of wood, place two Douglas fir 7-foot posts, in green condition, in an experimental pressure treating cylinder. Heat the contents of the cylinder to about 185° F., simultaneously drawing a vacuum until the pressure within the cylinder is about 15 to 25 inches of mercury. Maintain these conditions for about an 11 hour period to remove approximately 7 pounds of water per cubic foot of wood. Next, remove the solution from the cylinder and store in a separate tank. Apply 25 pounds per square inch air pressure to the interior of the cylinder. Fill the cylinder with the preservative solution while maintaining the internal pressure of 25 pounds per square inch. Next, apply pressure by means of a pump to raise the pressure within the cylinder to about 120 pounds per square inch and maintain this pressure for a period of about 3 hours at an average temperature of 160° F. This pressure forces the preservative solution into the posts.

It was desired to obtain a net retention of 8.0 pounds of preservative solution per cubic foot of wood. In order to accomplish this, inject approximately 10.0 pounds of preservative solution per cubic foot of wood. Next reduce the pressure within the cylinder to atmospheric pressure. Drain the solution from the cylinder. Apply a vacuum of 22 inches of mercury for a period of 45 minutes to strip excess preservative solution from the wood surface. Thus two posts are produced which are impregnated with the preservative solution and wax additive, having a net retention of 9.7 pounds of the applied composition per cubic foot of wood.

The following table gives data on numerous experiments employing different preservative solution compositions used for treating wood specimens. The data designated as the control were based on specimens treated with a 5% by weight solution of pentachlorophenol in an AWPA P9 oil without any wax. Treating solutions were made from samples of the control by adding 1% by weight of each of the waxes designated A through O in the table to each sample. Each treating solution designated A through O was used separately to treat four specific ponderosa pine specimens, each set of specimens being treated in only one of the solutions, according to the following procedure:

Four specimens of ponderosa pine were immersed as indicated above in each of the solutions, a vacuum applied to draw air outwardly from the wood specimens to permit the entry of the solutions. Next the pressure was raised to atmospheric pressure so that the increase in pressure would force the solution into the wood specimens. Thereafter the solution was drained from the treating vessel and a final vacuum applied to strip excess solution from the surface of the wood. Matched blocks of wood were employed and uniform pressures, temperatures, treating times and other conditions were employed in order that direct and reliable comparisons could be made on each set of specimens between test solutions or wax additives.

Each block (specimen) was weighed prior to treatment and after treatment to determine the amount of preservative solution retained therein. After the foregoing treatment to impregnate the blocks with solution, each block was suspended over a tared beaker in a chamber having controlled conditions. Next, the chamber was heated to raise the temperature from room temperature to 160° F. over a two-hour period to produce exudation of the solution from the wood. The heating was continued to raise the temperature from 160° F. to 180° F. so as to produce an average temperature in the center of the blocks of about 170° F. This three-hour heating cycle was repeated each day over a period of 20 days. At the end of this period, the tared vessel was weighed and the amount of exudate determined. In the case of compositions employing waxes A through O in the table, column 14 was computed by subtracting the percent exudation from the treatment with each wax preservative solution from the percent exudation of the control. This difference was divided by the percent exudation of the control and multiplied by 100 to determine the percent reduction in exudation.

Wood treating compositions containing waxes C, D, E, F, H, I, and K in the table are within the scope of the present invention. It will be noted from column 14 of the table that these waxes, when mixed to form preservative solutions according to the invention, produce a dramatic reduction in exudation from the wood specimens. Thus the useful life of the wood under service conditions is extended. Furthermore, the appearance of the wood is improved because of controlled exudation.

Table

Wax	Type[1]	(1) ASTM (D87) M.P. °F.	(2) Refractive index n_d^{100}	(3) Viscosity (wax) SSU at 210 °F.	(4) Pour point 1% wax solu. in AWPA P9 oil	(5) Wax necessary to form gel in AWPA P9 oil at 75° F., percent	(6) Average number carbon atoms	(7) Average molecular weight	(8) Urea reactable, percent	(9) Normal alkanes, percent	(10) Iso-alkanes, percent	(11) Mono-cyclo-alkanes, percent	(12) Dicyclo-alkanes, percent	(13) Mono-aromatic, percent	(14) Reduction in exudation from wood, percent
Control[2]					+7										
A	3	124.3	1.4184	36.3	+12	24.5	24	338		95.6	2.3	1.8	0.3		5.7
B	3	132.8	1.4212	39.3	+10	17.7	26	366		88.4	4.5	6.0	0.9	0.2	25.4
C	2	139.1	1.4239	41.5	+16	10.0	27	380		82.5	6.4	9.1	1.6	0.4	38.4
D	2	140.2	1.4244	41.1	+18	10.6	28	395		81.0	6.5	9.9	2.1	0.5	34.8
E	2	159.6	1.4305	51.4	+25	6.0	34	478		67.5	7.5	17.3	6.2	1.5	63.8
F	1	176.4	1.4413	77.8		11.5			50–90						
G	1	141.3	1.4435	80.0	+14	26.4			20–35						0
H	1	181.6		75.0		8.56			50–90						
I	1	181.6	1.4368	75.0	+15	8.56			50–90						49.7
J	1	166.6	1.4496	89.8		15.60			45–65						
K	1	170.0	1.4396	77.0	+11	10.00			45–65						50.8
L	1	160.0	1.4427	91.1	+10	19.5			20–40						39.2
M	4	200.0			+10	10.0									39.5
N	5	190.0			+10	12.7									35.0
O	6	186.0		270.0	+13	20.0									29.3

[1] 1=residual-microcrystalline, 2=distillate intermediate paraffin, 3=distillate-paraffin, 4=synthetic-Fisher Tropsch-iso paraffin, 5=vegetable, 6=mineral-lignite extract.

[2] No wax—only the mixture of pentachlorophenol and AWPA P9 oil.

In the attached drawing, there is shown a graph based on some of the data disclosed in the table. In the graph, percent reduction in exudation, column 14 of the table,

is plotted as ordinate and percent wax required to produce a gel, column 5 of table, as abscissa. From the data plotted in this graph, it will be seen that the lower the percentage of wax required to produce a gel, the greater the reduction in exudation. It would appear that this provides a procedure for identifying waxes suitable for the invention. That is, where a low percentage of a wax is required to produce a gel, such a wax merits further investigation to determine if it comes within the scope of this patent disclosure.

Thus it will be seen that the invention provides a composition for preserving wood having increased efficiency and a method of applying same to wood. The invention provides an increased useful life of wood treated with the solution, which results in an economy of treatment.

While a present preferred embodiment of the invention has been described, it will be understood that various modifications of the invention can be produced within the scope of the appended claims.

We claim:

1. A composition for preserving wood which comprises a preservative for combating biological attack on wood, and a wax having an average molecular weight not exceeding 600, a viscosity between about 40 and 80 Saybolt Universal seconds at 210° F., said wax being crystallizable into crystalline forms capable of forming gels and holding the preservative within the crystalline structure.

2. A composition for preserving wood which comprises a toxicant for combating biological attack on wood, a hydrocarbon carrier, and a wax having an average molecular weight between about 350 and 600 and a viscosity between about 40 to 80 Saybolt Universal seconds at 210° F., said wax being crystallizable to form a crystalline lattice capable of adsorbing said hydrocarbon carrier to form a gelled mixture.

3. The invention as set forth in claim 2 wherein said wax is present in about 1% to 2% by weight of the entire mixture.

4. A method of preserving wood comprising the steps of mixing a preservative for combating biological attack on wood with a wax having an average molecular weight of not exceeding about 600, and a viscosity between about 40 and 80 Saybolt Universal seconds at 210° F., and impregnating wood with said mixture.

5. The invention as set forth in claim 4 wherein said preservative is of the oil-borne type.

6. The invention as set forth in claim 4 wherein said preservative is of the oil-type preservative.

7. A process for preserving wood comprising the steps of mixing a toxicant, a petroleum hydrocarbon carrier, and not more than 10% of a wax having an average molecular weight from about 350 to about 600 and a viscosity between about 40 and 80 Saybolt Universal seconds, and, impregnating wood with said mixture.

8. A composition for preserving wood which comprises a wood preservative and wax means having a molecular configuration to reduce the motility of the preservative in the liquid state, said wax means having a crystal lattice in the solid state to form a gel of the preservative and thus reduce the motility of the preservative.

9. A composition for preserving wood which comprises a wood preservative and a wax having a molecular configuration to reduce the motility of the preservative in the liquid state, said wax having a crystal lattice in the solid state to form a gel of the preservative and thus reduce the motility of the preservative, said wax having a viscosity, determined at 210 degrees Fahrenheit, between about 40 and about 80 Saybolt Universal seconds.

10. A composition for preserving wood which comprises a preservative for combating biological attack on wood; and a wax having an average molecular weight not exceeding 600, a viscosity, measured at 210 degrees Fahrenheit, between about 40 and about 80 Saybolt Universal seconds, a refractice index n_d^{100} between about 1.4230 and about 1.4415, said wax being crystallizable into crystalline forms capable of forming gels and holding the preservative within the crystalline structure, said wax melting between about 135 and 185 degrees Fahrenheit, said wax being present in an amount not greater than about 10 percent by weight of the composition.

11. A composition for preserving wood which comprises pentachlorophenol; a petroleum hydrocarbon carrier; and a wax having an average molecular weight between about 350 and about 600, a viscosity between about 40 and about 80 Saybolt Universal seconds, measured at 210 degrees Fahrenheit, said wax being crystallizable to form a crystalline lattice capable of adsorbing said petroleum hydrocarbon carrier to form a gelled mixture.

12. A composition for preserving wood which comprises a preservative for combating biological attack on wood; and an intermediate paraffin distillate wax that melts at about 159.6 degrees Fahrenheit, has a refractive index n_d^{100} of about 1.4305, and has a viscosity, measured at about 210 degrees Fahrenheit, of about 51.4 Saybolt Universal seconds, said wax containing approximately 67 percent normal alkanes, 7.5 percent isoalkanes and about 17 percent monocycloalkanes.

13. In the process of preserving wood which includes the steps of applying a partial vacuum to wood; subsequently applying a wood preserving composition to the wood under a positive pressure to force the composition into the wood; the improvement which comprises said composition including a preservative and wax means having a molecular configuration to reduce the motility of the preservative in the liquid state, said wax means having a crystal lattice in the solid state to form a gel of the preservative and thus reduce the motility of the preservative.

14. In a process of preserving wood which includes the steps of applying a partial vacuum to wood and subsequently impregnating a wood preserving composition into the wood under a positive pressure forcing the composition into the wood; the improvement wherein said wood preserving composition comprises a preservative and a wax having a molecular configuration to reduce the motility of the preservative in the liquid state, said wax having a crystal lattice in the solid state to form a gel of the preservative and thus reduce the motility of the preservative, said wax having a viscosity, determined at about 210 degrees Fahrenheit, between about 40 and about 80 Saybolt Universal seconds, said wax being present in an amount sufficient to reduce said motility but not greater than about 10 percent by weight of the composition.

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