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WOOD PRESERVATIVE GREASE

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This invention relates to a method and composition for preservation of wood, particle board, hard board, and related structural fibrous materials, which method and composition are designed to increase the performance in wood preservation, simplify the application to the wood and improve the overall results obtained.

There are many methods of applying preservatives to wood prior to the time the wood is put in use. The composition of the instant invention may have utility in this respect, although it is intended primarily for use on wood already in place, such as poles, posts, sills, toe plates, stair risers, etc.; and the instant invention will be described primarily in connection with this use.

One of the chief uses for the present invention is in extending the usable life of poles already in line. Some of these poles may have been in place many years and the application of the instant composition thereto will add extra years of life to the pole. Those poles which were poorly pretreated or pretreated with inferior preservatives may be again treated, while in place, with the instant composition along the pole. Poles which are removed from one location to be set in another, usually at a different depth, can be advantageously treated with the composition of the instant invention.

The instant composition is a grease, or a grease-like material, which may be applied by hand spreading, by a mechanical grease gun, or on a paper or plastic film or bandage. On poles it is applied at the ground line area, where rot is most likely to occur. There are at present several wood preservative greases known and in use. Each of these leaves something to be desired, however. The composition of the instant invention overcomes most of the difficulties or problems encountered in the use of the prior art compositions. For example, in British Patent No. 699,207 (published November 4, 1953) reference is made to a grease-like substance which can be applied to wooden objects along the ground line, and the greases described by the British patent are oil base materials thickened with soaps such as aluminum or calcium stearate. Such greases are difficult to manufacture; and they have the disadvantage that the number of oils that can be used as the solvent for the active fungicide is limited. Also, such greases have a tendency to bleed at room temperature and at elevated temperatures frequently encountered in storage and in the field during summer. In addition, the preservative portion of such greases sometimes does not penetrate the wood well enough.

Other preservative gels or greases now on the market have various disadvantages which have been overcome by the product of the instant invention. These disadvantages include poor resistance to water, inadequate penetration of the wood, difficulty of application, high cost, doubtful preservative value, bleeding of the fluid, and a tendency to change in consistency.

In the instant invention, an organic wood preservative liquid is thickened with a compound of bentonite and an organic-nitrogen base, in an amount sufficient to

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impart a grease-like consistency to the composition, and this grease-like composition is applied to wood. It has been found that the thickening compound of bentonite and an organic-nitrogen base has unique properties in that it is capable of imparting the desired consistency to the grease-like composition, while permitting penetration of the wood preservative to a sufficient extent into wood to which the grease is applied. This thickening compound produces a distinctly superior grease in that bleeding is substantially eliminated. Also, the material is easily compounded and applied.

It is, therefore, an important object to provide an improved composition and method for wood preservation.

It is a further object of the instant invention to provide an improved grease-like wood-preserving composition that does not tend to bleed.

It is another object of the instant invention to provide an improved grease-like composition that is easily applied to wood for the preservation thereof, comprising an organic wood preservative liquid and a thickening compound of bentonite and an organic-nitrogen base.

Yet another object of the instant invention is to provide an improved method of preserving wood that comprises milling or subjecting to shear a composition comprising an organic wood preservative liquid and a thickening compound of bentonite and an organic-nitrogen base (with one of many standard colloid mills or homogenizers), and applying the composition to the wood.

Other and further objects, features and advantages of the present invention will become apparent to those skilled in the art from the following detailed disclosure thereof.

The invention consists in a wood preservative composition comprising an organic wood-preservative liquid, and a compound of bentonite and an organic-nitrogen base in an amount sufficient to impart a grease-like consistency to said composition.

The organic wood-preservative liquid used in the practice of the instant invention acts as a diluent for the composition and additionally functions as a spreading agent for the final composition. This liquid may be a solution of a wood preservative in an organic liquid carrier (such as a solution of pentachlorophenol in an aromatic petroleum oil or a solution of copper naphthenate in mineral spirits) or it may be a liquid preservative per se as in the case of creosote. By "creosote" is meant materials so designated by chemists which are, in fact, organic mixtures derived from coal tar or wood tar and prepared commercially as wood-preserving liquids, examples of which include coal tar creosote, coke oven tar creosote, water gas tar creosote, petroleum oil creosote, wood creosote, and mixtures thereof. Each of such creosotes may be used in the practice of the instant invention and they may be used with or without additional solvents or carriers, such as petroleum oil; or they may be used with additional preservatives such as copper naphthenate or pentachlorophenol.

The amount of organic wood preservative liquid in the final composition of the invention depends to a substantial extent upon the viscosity of this liquid and also the nature of the molecules composing the liquid; and it may range from about 60% for other thin liquids to as much as about 90-94% for thick liquids. (As used herein, the terms "percent" and "parts" mean percent and parts by weight, unless otherwise designated.)

As mentioned, the organic wood-preservative liquid used in the practice of the invention may be a liquid preservative per se such as creosote. Also, it may be a preservative such as a polyvalent metal naphthenate such as zinc or copper naphthenate dissolved in a suitable (preferably non-polar) solvent, such as a mineral oil. The preferred organic wood preservative liquid of this

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type is a solution of copper naphthenate in a mineral oil; and the resulting grease-like composition preferably contains at least 2% of the metal (Cu) and preferably 10% to 30% of the copper naphthenate is used.

The organic fungicide found to be most desirable for use in the practice of the instant invention is pentachlorophenol. The amount of pentachlorophenol in the final grease-like composition should be at least about 2% and is preferably within the range of 5% to 30% of the weight of the composition.

In the practice of the instant invention, the organic wood-preservative liquid is first provided (in the form of creosote) or prepared by dissolving the organic fungicide in a suitable solvent therefor such as a mineral oil. Then the bentonite compound is added and stirred into the liquid. Next, the relatively small quantity of methanol, acetone or other organic polar solvent used as a dispersing agent is added. Then, any suitable inorganic filler material to be employed is added. The resulting material is a liquid slurry, which is then passed through a homogenizer or colloid mill wherein it is subjected to 1000 to 8000 pounds per square inch of shear and the slurry is converted to a grease-like gel. Inorganic fungicide, if used, is preferably added next.

The foregoing procedure is carried out using the following formulations and the results indicated in connection with each of these examples are the results obtained:

EXAMPLE 1

	Percent
Diocetadecyldimethyl ammonium bentonite	10.0
Methanol	0.7
Shell Medium Aromatic oil	79.3
Pentachlorophenol	10.0

ASTM penetration: 400; bleeding resistance and resistance to water good; and penetration into wood good.

EXAMPLE 2

	Percent
Dicetyldimethyl ammonium bentonite	10
Aromatic HB oil	78.6
Pentachlorophenol	10
Acetone	1.4

ASTM penetration: 294; bleeding resistance excellent, resistance to water satisfactory, penetration into wood satisfactory.

The pentachlorophenol is incorporated in a wood-preservative liquid first and the thickening agent is added subsequently. The solvent used for pentachlorophenol in the liquid is preferably a mineral oil; and the best results are obtained using an aromatic petroleum oil or sometimes called an aromatic gas oil or a heavy or light recycle oil. A preferred oil is a heavy recycle oil having a boiling point of about 600-760° F. Other oils which may also be used include cracking stocks, slurry oils, heavy aromatic gas oils and medium aromatic gas oils, examples being Amoco heavy thermal side cut No. 105, Amoco No. 102E oil, Tidewater No. 200 diesel oil, Tidewater CGB oil, Denver No. 3 oil, Magnolia lube oil extract No. 465E, Aromatic HB oil, Heavy Aromatic Naphtha, Shell Medium Aromatic oil, and Deep Rock heavy cycle oil. Other solvents for the organic fungicides here mentioned include naphthenic oils, petroleum tar gas oil, linseed oil, xylene, mineral spirits and butyl Cellosolve. The last mentioned solvent will dissolve up to 60% pentachlorophenol and can be used to make a grease containing 50% pentachlorophenol for special purposes. An aromatic petroleum oil such as Aromatic HB is ordinarily preferred for use in the practice of the instant invention with pentachlorophenol, because of proper viscosity and good solvent power for pentachlorophenol.

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The thickening agent employed in the practice of the instant invention for obtaining a grease-like consistency in the final composition is a compound of bentonite and an organic-nitrogen base. Bentonite compounds are composed of a montmorillonite mineral in which at least a part of the cation content of the mineral has been replaced by an organic-nitrogen base. Clays that swell at least to some extent on being contacted with water and contain as a primary constituent a mineral of the group known as montmorillonites are generally referred to as bentonite. Such clays, which contain exchangeable alkali metal atoms either naturally or after treatment, constitute the raw materials employed in making the bentonite-organic base compounds used in the practice of the instant invention. The bentonite-organic base compounds are preferably prepared as described in U.S. Patent No. 2,033,856, issued March 10, 1936, as compounds of bentonite with organic bases of sufficient alkalinity to be titratable with mineral acids, which compounds are made by bringing together the bentonite and the organic-nitrogen base in the presence of aqueous mineral acid to effect base exchange. The organic-nitrogen bases include cyclic, aliphatic and heterocyclic amines, such as decyl amine, dodecyl amine, tetra decyl amine, hexadecyl amine, octadecyl amine, hexadecyl ammonium acetate, octadecyl ammonium acetate, dimethyl dioctyl ammonium acetate, dimethyl didodecyl ammonium acetate, dimethyl dodecyl hexadecyl ammonium acetate, dimethyl diacetate ammonium acetate, dimethyl dodecyl hexadecyl ammonium acetate, dimethyl diacetate ammonium acetate, dimethyl hexadecyl octadecyl ammonium acetate, dimethyl dioctadecyl ammonium acetate, and the corresponding chlorides and quaternary ammonium chlorides. The preferred bentonite compounds are prepared from quaternary ammonium compounds in which the N-substituents are aliphatic groups containing at least one alkyl group with a total of at least ten to twelve carbon atoms. When aliphatic amines are used they preferably contain at least one alkyl group containing at least ten to twelve carbon atoms.

Although the bentonite compounds are particularly useful in the practice of the instant invention as thickeners for the final grease composition, it will be noted that some of the bentonite compound may be replaced by inert inorganic fillers such as finely divided silica, diatomaceous earth, and finely divided calcium silicate. In general, the total amount of bentonite compound and inorganic filler (if any is used) should be such as to effect the desired thickening of the composition and this amount may range from about 5% to about 40% of the final composition. The weight ratio of bentonite compound to the inorganic filler may range from 1:10 to 10:1.

In addition, a dispersing agent is preferably employed with the bentonite compound. The amount of dispersing agent employed is 0.5% to 5% of the final grease composition, or 5 to 25% of the amount of bentonite compound used. The dispersing agent is an organic polar solvent such as methanol, acetone or the like (preferably a low molecular weight alcohol, ketone or ester containing not more than about 4 carbon atoms such as methanol, ethanol, isopropanol, butanol, acetone, diethyl ketone, methyl acetate, ethyl acetate, etc.). In the practice of the instant invention the dispersing agent is added to the composition after the bentonite compound, but before any inert filler materials are added.

Also, inorganic fungicides such as sodium, potassium or ammonium fluorides may be added to the instant composition. Such materials are also added with the inorganic filler (if any), after the addition of the other ingredients. Such materials may be employed in amounts ranging from 1 to 20%.

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EXAMPLE 3

	Percent
Dicetyldimethyl ammonium bentonite	7.5
Aromatic HB oil	81.8
Methanol	0.7
Pentachlorophenol	10.0

ASTM penetration: 312, all other properties excellent.

EXAMPLE 4

Dicetyldimethyl ammonium bentonite	7.0
Heavy Aromatic Naphtha	82.3
Methanol	0.7
Pentachlorophenol	10.0

ASTM penetration: 400; slight but unobjectionable bleeding, all other properties excellent.

EXAMPLE 5

Dicetyldimethyl ammonium bentonite	3.5
Aromatic HB oil	72.5
Methanol	1.0
Pentachlorophenol	10.0
Silica (HI-SIL 233)	13.0

ASTM penetration: 240; all other properties excellent.

EXAMPLE 6

Dicetyldimethyl ammonium bentonite	10.7
Coal tar creosote	87.9
Methanol	1.4

ASTM penetration: 400; all other properties good.

EXAMPLE 7

Dicetyldimethyl ammonium bentonite	10.0
Coal tar creosote	44.3
Heavy Aromatic Naphtha	40.0
Pentachlorophenol	4.3
Methanol	1.4

ASTM penetration: 400; all other properties excellent.

EXAMPLE 8

Dicetyldimethyl ammonium bentonite	7.0
Tidewater diesel oil	86.1
Methanol	2.4
Pentachlorophenol	4.5

ASTM penetration: 400; all other properties good.

EXAMPLE 9

Dicetyldimethyl ammonium bentonite	14.8
Copper naphthenate	27.9
Mineral spirits	55.9
Methanol	1.4

ASTM penetration: 340; all other properties good.

EXAMPLE 10

Dicetyldimethyl ammonium bentonite	7.0
Aromatic HB	78.6
Pentachlorophenol	8.7
Methanol	0.7
Sodium fluoride	5.0

ASTM penetration: 400; all other properties good.

EXAMPLE 11

Dicetyldimethyl ammonium bentonite	3.0
Aromatic HB	77.7
Methanol	0.7
Pentachlorophenol	8.6
Calcium silicate (Microcel-E)	10.0

ASTM penetration: 311; all other properties good.

EXAMPLE 12

Octadecyl amine bentonite	12.5
Aromatic HB	74.2
Pentachlorophenol	8.3
Methanol	5.0

ASTM penetration: 331; all other properties good.

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EXAMPLE 13

	Percent
Dicetyldimethyl ammonium bentonite	7.0
Deep Rock heavy cycle	83.1
Pentachlorophenol	9.2
Methanol	0.7

ASTM penetration: 400; all other properties good.

The National Lubricating Grease Institute developed and adopted a consistency classification in 1941. This classification, which is based on penetrations obtained by ASTM Method D 217, is shown in Table 21.1. When the Federal Government put in effect a tax on lubricating oils (see Regulation 44, Section 314.40, Use of Terms) the above method of test was recognized, as can be seen from the following quotation: "The term 'lubricating oils' does not include products of the type commonly known as grease. Oleaginous substances which are classed as grease and which contain oil are not subject to the tax when of a worked consistency of less than 390 penetration units, or an unworked consistency of less than 360 penetration units, by the method of test of the American Society for Testing Materials D-217-33-%."

Table 21.1.—NLGI lubricating grease consistency classification

Consistency number:	ASTM worked penetration at 77° F.
0	355 to 385
1	310 to 340
2	265 to 295
3	220 to 250
4	175 to 205
5	130 to 160
6	85 to 115

ASTM Method D217-52T does not provide for tests of lubricating greases softer than those having a penetration of about 400, and some of the greases of the invention have a penetration of 400 or slightly over; but for the purposes of the invention "greases" have about 400 or less penetration.

EXAMPLE 14

	Percent
Dicetyldimethyl ammonium bentonite	3
Silica (Cabo-Sil)	3
Aromatic HB oil	83.3
Methanol	0.7
Pentachlorophenol	10.0

ASTM penetration: 400; all other properties excellent.

EXAMPLE 15

	Percent
Dicetyldimethyl ammonium bentonite	7.9
Coal Tar Creosote	16.0
Sodium fluoride	12.0
Aromatic HB oil	52.2
Pentachlorophenol	14.4
Methanol	1.2

ASTM penetration: 345; all other properties good.

EXAMPLE 16

	Percent
Dicetyldimethyl ammonium bentonite	13.2
Pentachlorophenol	43.4
Butyl cellosolve	43.4

ASTM penetration: 300; all other properties excellent.

As a specific example, using the previously quoted formulation of Example 1, pentachlorophenol (10 parts) is dissolved in Shell medium aromatic oil (79.3 parts). Next, dioctadecyldimethyl ammonium bentonite (10 parts) is stirred into the solution of pentachlorophenol in

Shell medium aromatic oil. Then, methanol (0.7 part) is also stirred into the resulting slurry of dioctadecyldimethyl ammonium bentonite in the Shell medium aromatic oil solution of pentachlorophenol, which material is at this stage a relatively free flowing liquid (slurry). The material is then passed through a homogenizer (wherein it is subjected to about 2000 pounds per square inch of shear) and the resulting material obtained from the homogenizer is a grease-like gel having the properties described in connection with Example 1. The same procedure is employed in preparing each of the other formulations set forth herein, to obtain the results specified.

The grease of Example 1 is applied to poles in line (along the ground line) and found to effectively preserve the wood. In areas of rainfall of about 15 inches or more a year, this grease-like material is applied to an area on the pole extending from about 6 inches above the ground line to about 20 inches below. In arid regions, the grease-like material is applied from approximately the ground line to as much as 3 or more feet below the ground line. The instant grease-like material is also applied to the exposed tops of poles and found to be an effective preservative in this respect also. Each of the aforementioned examples of grease-like material are used in substantially the manner just described to obtain effective wood preservation and prolong the life of poles in the line.

Results also indicate that adequate penetration using the instant greases is obtained in seasoned wood as well as wood having a moisture content of as much as 40%. For example, pole stubs of southern yellow pine placed in the soil are found to have an equilibrium moisture content at the ground line of 35 to 40% by weight of the dry wood; and these pole stubs are effectively preserved by the application of the instant grease-like material. It should be mentioned that, in such cases where the moisture content is high, a preferred formulation is that of Example 10 which specifies the use of a water-soluble preservative salt such as sodium fluoride in the grease-like composition.

It will be understood that modifications and variations may be effected without departing from the spirit and scope of the novel concepts of the present invention.

I claim as my invention:

1. A wood preservative composition comprising at least 2% of pentachlorophenol, 50% to 95% of a mineral oil solvent for the pentachlorophenol, and a compound of bentonite and an organic-nitrogen base in an amount sufficient to impart a grease-like consistency to said composition said compound being a compound of bentonite with an organic base of sufficient alkalinity to be titratable with mineral acids, which compound is made by bringing together bentonite and such organic base in the presence of water.

2. A wood preservative composition comprising at least 2% of pentachlorophenol, 50% to 95% of a mineral oil solvent for the pentachlorophenol, 0.5% to 5% of an organic polar solvent, and a compound of bentonite and an organic-nitrogen base in an amount sufficient to impart a grease-like consistency to said composition, said compound being a compound of bentonite with an organic base of sufficient alkalinity to be titratable with mineral acids, which compound is made by bringing together bentonite and such organic base in the presence of water.

3. A wood preservative composition comprising at least

2% of pentachlorophenol, 50% to 95% of a mineral oil solvent for the pentachlorophenol, a combination of (a) an inert inorganic filler and (b) a compound of bentonite and an organic-nitrogen base in an amount sufficient to impart a grease-like consistency to said composition, the weight ratio of (a):(b) ranging from 1:10 to 10:1 said compound being a compound of bentonite with an organic base of sufficient alkalinity to be titratable with mineral acids, which compound is made by bringing together bentonite and such organic base in the presence of water.

4. A wood preservative composition comprising at least 2% of pentachlorophenol, 50% to 95% of a mineral oil solvent for the pentachlorophenol, 0.5% to 5% of an organic polar solvent, a combination of (a) an inert inorganic filler and (b) a compound of bentonite and an organic-nitrogen base in an amount sufficient to impart a grease-like consistency to said composition, the weight ratio of (a):(b) ranging from 1:10 to 10:1, said compound being a compound of bentonite with an organic base of sufficient alkalinity to be titratable with mineral acids, which compound is made by bringing together bentonite and such organic base in the presence of water.

5. A wood preservative composition comprising at least 2% of pentachlorophenol, 50% to 95% of a mineral oil solvent for the pentachlorophenol, and a combination of (a) silica and (b) a compound of bentonite and an organic-nitrogen base in an amount sufficient to impart a grease-like consistency to said composition, the weight ratio of (a):(b) ranging from 1:10 to 10:1 said compound being a compound of bentonite with an organic base of sufficient alkalinity to be titratable with mineral acids, which compound is made by bringing together bentonite and such organic base in the presence of water.

6. A wood preservative composition having the following formulation:

	Percent
Dicetyldimethyl ammonium bentonite	7.5
Aromatic HB oil	81.8
Methanol	0.7
Pentachlorophenol	10.0

7. A wood preservative composition having the following formulation:

	Percent
Dicetyldimethyl ammonium bentonite	3.5
Aromatic HB oil	72.5
Methanol	1.0
Pentachlorophenol	10.0
Silica	13.0

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