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WOOD TREATMENT

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The instant invention relates to the treatment of wood, and more particularly, to anti-blooming agents for use in wood preservation and improved methods of preserving wood by the use of such anti-blooming agents.

Although various compounds are known for the preservation of wood, pentachlorophenol has met with great commercial acceptance. In the usual wood preserving process, pentachlorophenol dissolved in a suitable solvent is brought into contact with the wood, as by dipping the wood in the solution, and the wood is then removed and dried. The pentachlorophenol, of course, penetrates into the wood and to a substantial extent remains impregnated in the wood after the solvent has been dried or removed. One difficulty, however, is that there is a tendency for some of the pentachlorophenol to leach back out of the wood during the drying process and, after the wood is dried, there appears a light colored crystalline coating on the surface of the wood which may be easily rubbed off (and is thus ineffective for wood preservation purposes). This results in an undesirable appearance as well as an undesirable loss of material. This phenomenon is referred to as "blooming" in the industry.

The exact causes of blooming are not fully understood in the industry, but the problem has persisted for a number of years. Various "anti-blooming" agents may have been suggested from time to time but each has left something to be desired.

The instant invention resides in the discovery that certain compounds, which will be referred to generally hereinafter as "amido" compounds (but which are described in detail in the specification) may be used in conjunction with pentachlorophenol, in solution, during treatment or impregnation of the wood so as to materially reduce or substantially eliminate blooming. These amido compounds which have been found to have unique anti-blooming properties include only a very specific class of compounds. The peculiar manner in which these antiblooming agents apparently cooperate with pentachlorophenol cannot be fully understood, but the unusual results speak for themselves, as will be shown hereinafter.

It is, therefore, an important object of the instant invention to provide a unique anti-blooming agent for the treatment of wood; and to provide an improved wood preservation process.

It is a further object of the instant invention to provide a wood treating composition comprising pentachlorophenol and an N-dialkylaminoalkyleneamide or salt thereof; and it is yet another object of the instant invention to provide an improved method of treating wood which comprises contacting the wood with said composition.

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 instant invention consists in a composition comprising pentachlorophenol and an amido compound, dissolved in a common solvent, the concentration of the pentachlorophenol being 1% to 30% and the concentration of the amido compound being ½ to 10 times the pentachlorophenol concentration, said amido compound being selected from compounds having the formulae

$$RCO—NR'—A—NR_2''$$

and

$$RCO—NR'—A—NR_2''.HO—OCR$$

wherein RCO is an acyl group derived from a carboxylic acid selected from C_{12}–C_{20} fatty acids and rosin acids, A is a C_2–C_6 alkylene group, R' is a radical selected from the group consisting of H and C_1–C_4 alkyl radicals, and R'' is a C_1–C_4 alkyl radical; and a method of treating wood which comprises contacting the wood with said composition and then drying the wood.

Although mono and polychlorophenols are generally known to have wood preserving properties, and particularly chloro-2-phenylphenol, tetrachlorophenol as well as pentachlorophenol (any of such mono and/or polychlorophenols may be used in the practice of the instant invention), pentachlorophenol, tetrachlorophenol and chloro-2-phenylphenol are the members of this group which have met with the most general commercial acceptance. Actually, commercial pentachlorophenol (which is used in the following examples) is predominantly pentachlorophenol although it contains an appreciable amount of tetrachlorophenol also. The commercial analysis is 83% pentachlorophenol, 12% tetrachlorophenol and 5% inerts.

The instant invention does not involve a departure from the use of previously employed pentachlorophenol solutions or the previously employed process steps for the use of such solutions, with the exception that the instant anti-blooming agent is included in the solution and is use in conjunction with the pentachlorophenol. Pentachlorophenol solutions for this commercial purpose are well known in the art. The solutions may have concentrations within the range of 1% to 30% pentachlorophenol. (As used herein, the terms "parts" and "percent" mean parts and percent by weight unless otherwise indicated.) Preferably, the pentachlorophenol concentration is within the range of about 3% to about 8%, and the best results are ordinarily obtained using 5% pentachlorophenol in the solution.

The solvents used in commercial practice are all relatively slowly volatile solvents and, of course, since such solvents are lost during the drying process to a substantial extent, economic considerations are involved. In general, hydrocarbon solvents are employed. Preferred solvents include mineral spirits, xylene, etc. boiling within the range of 130° C. to about 200° C. In this way bath temperatures of substantially greater than room temperature may be used so as to obtain better solution of the pentachlorophenol and the amido compound and more effective and more rapid penetration of the wood, although bath temperatures for the solution (in which the wood is usually dipped) may range from 0° C. to as much as about 40° C. (in open treating vessels).

The concentration of the amido compound used in the practice of the instant invention depends to a certain extent upon the concentration of the pentachlorophenol and, in general, the concentration of the amido compound may range from a minimum of about ½ to a maximum of about 10 times the pentachlorophenol concentration. Expressed in other terms, the amido compound concentration may range from a minimum effective amount of about ½% to a maximum effective amount of perhaps 30%, the total of the pentachlorophenol and amido compound concentrations should not, ordinarily, exceed about 50% in order to maintain effective solution of both ingredients in the solvent, at usual working temperatures. In the preferred ranges of pentachlorophenol concentration it is generally preferable to employ an amido compound concentration that is substantially equal to the

pentachlorophenol concentration, and it has been found that the best results are obtained using substantially a 5% pentachlorophenol concentration and substantially an equal amido compound concentration.

As indicated previously, the amido compound used in the practice of the invention is an N-dialkylaminoalkyleneamide or salt thereof. In each case, the amide is an amide of a high molecular weight acid and the salt is a salt of a high molecular weight organic acid. Certain derivatives of 3-dimethylaminopropylamine and fatty acids and/or rosin acids are preferred.

Suitable N-dialkylaminoalkylamide compounds of the indicated type are illustrated in the following formula:

$$RCO—NR'—A—NR_2''$$

where RCO— represents an acyl group derived from a carboxylic acid, i. e., from a fatty acid containing 12 to 20 carbon atoms or from a rosin acid; A represents an alkylene group containing 2 to 6 carbon atoms; R' represents a hydrogen atom or an alkyl group containing 1 to 4 carbon atoms and R'' represents an alkyl group containing 1 to 4 carbon atoms, where each R'' may be the same or different.

Particularly effective and desirable compounds of the class of compounds of this invention are:

$$C_{17}H_{33}CONHCH_2CH_2CH_2N(CH_3)_2$$

(N-dimethylaminopropyl oleamide) and

$$C_{17}H_{33}CONHCH_2CH_2N(CH_3)_2$$

(N-dimethylaminoethyl oleamide). Among the other particular compounds of this class of compounds are: $C_{11}H_{23}CONHCH_2CH_2N(C_4H_9)_2$ (N - dibutylaminoethyl lauramide),

$$C_{17}H_{31}CON(CH_3)C_6H_{12}N(C_2H_5)_2$$

(N-methyl-N-diethylaminohexyl linoleamide) and

$$C_{17}H_{33}CON(C_3H_7)CH_2CH_2N(CH_3)C_3H_7$$

(N-isopropyl-N-methylisopropylaminoethyl oleamide).

The invention also relates to carboxylic acid salts of N-dialkylaminoalkylamides as represented by the formula:

$$RCO—NR'—A—NR''_2.HO—OCR$$

wherein RCO—, R', R'', and A represent the same groups as described previously. The RCO— in these types of compounds may be the same or different.

The following are particularly effective and desirable examples of this class of anti-blooming agents:

$$C_{17}H_{33}CONHCH_2CH_2N(CH_3)_2.HOOCR$$

(rosin acid salt of N-dimethylaminoethyl oleamide) and

$$C_{17}H_{33}CONHCH_2CH_2CH_2N(CH_3)_2.HOOCR$$

(rosin acid salt of N-dimethylaminopropyl oleamide). Among the other particular compounds of this class of compounds are:

$$C_{11}H_{23}CONHCH_2CH_2CH_2N(C_3H_7)_2.HOOCC_{17}H_{33}$$

(N-diisopropylaminopropyl lauramide oleate),

$$C_{17}H_{31}CON(CH_3)CH_2CH_2N(CH_3)C_3H_7.HOOCC_{11}H_{23}$$

(N - methyl - N - methylisopropylaminoethyl linoleamide laurate), and

$$C_{17}H_{33}CON(C_4H_9)C_4H_8N(C_4H_9)_2.HOOCC_{17}H_{33}$$

(N-butyl-N-dibutylaminobutyl oleamide oleate).

It should be noted that both types of compounds contain a tertiary amine group and an amide group and it is this particular structure which is believed to impart to the compounds their desired characteristics.

In preparing the N-dialkylaminoalkylamides and their carboxylic acid salts, organic acids are reacted with dialkylaminoalkylamines. Representatives of the class of these amines are:

$$(CH_3)_2NCH_2CH_2CH_2NH_2$$

(3-dimethylaminopropylamine),

$$(CH_3)_2NCH_2CH_2NH_2$$

(dimethylaminoethylamine),

$$(CH_3)_2NCH_2CH_2NHCH_3$$

(dimethylaminoethyl methylamine),

$$(C_4H_9)_2NCH_2CH_2CH_2CH_2NHC_4H_9$$

(4-dibutylaminobutyl butylamine), and

$$C_3H_7(CH_3)NCH_2CH_2NHCH_3$$

(methylisopropylaminoethyl methylamine).

Representative of the organic acids that may be used are oleic, linoleic, linolenic, lauric, palmitic, stearic, rosin acids, abietic acid, the mixed acids of tall oil, or mixtures of any of these acids. Triglycerides, such as vegetable and fish oils and animal fats, may be used as ingredients to supply the fatty acids required in carrying out the chemical preparation.

Commercially available acids (which are referred to by letter hereinafter) that are used in the practice of the invention include:

Acid A: (FA No. 1)—Distilled tall oil containing 90–93% fatty acids, 3–5% rosin acids.

Acid B: (Acintol D)—Distilled tall oil containing 30–34% rosin acids, 63–67% fatty acids.

Acid C: (Facoil GN)—Refined tall oil containing 54% fatty acids, 37% rosin acids.

Acid D: (Acintol C)—Crude tall oil containing 40–44% rosin acids, 47–52% fatty acids.

Acid E: (Acintol R)—Tall oil rosin containing 0–3% fatty acids.

Acid F: (Stearic acid)—Rubber grade—45% stearic, 55% palmitic.

Acid G: (TBO)—Tallow black oil—88% free fatty acids (26% palmitic, 62% stearic).

In preparing the N-dialkylaminoalkylamides, one mol of organic acid and one mol of dialkylaminoalkylamine are mixed together and heated at 100–200° C. in a suitable container with suitable agitation. Upon mixing, the amine salt of the organic acid is formed and during the heating process the salt is molecularly dehydrated forming one mol of the N-dialkylaminoalkylamide with the elimination of one mol of water, as follows:

Reaction on mixing:

$$RCOOH+NHR'—A—NR''_2 \rightarrow RCOOH.NHR'—A—NR''_2 \quad (1)$$

and

$$RCOOH+NHR'—A—NR''_2 \rightarrow RCOOH.NR''_2—A—NHR' \quad (2)$$

Reaction upon heating:

$$(1) \rightarrow RCO—NR'—A—NR''_2+H_2O$$

$$(2) \rightarrow (1) \rightarrow RCO—NR'—A—NR''_2+H_2O$$

The course of the reaction and its completion may be followed by noting the diminishing proportion of carboxylic acid by titration with standard alkali and the diminishing proportion of titratable amine with standard acid. At the completion of the reaction there is substantially no organic acid present and the titratable amine has been reduced to substantially one-half of its original value. The measurement of the eliminated water may also be useful as a guide in determining the completion of the reaction.

In preparing the carboxylic acid salts of the N-dialkylaminoalkylamides, equimolecular proportions of the N-dialkylaminoalkylamide and the organic acid are

mixed together at any suitable temperature at which the components are liquids:

$$RCO—NR'—A—NR''_2+RCOOH\rightarrow RCO—NR'—A—NR''_2.HOOCR$$

Alternatively, all reactions may be carried out concurrently by mixing one mol of dialkylaminoalkylamine and two mols of organic acid and reacting the mixture at 100–200° C. in a suitable container with suitable agitation:

$$2RCOOH+NHR'—A—NR''_2\rightarrow RCOOH.NHR'—A—NR''_2HOOCR \quad (1)$$

$$(1)\rightarrow RCO—NR'—A—NR''_2.HOOCR+H_2O$$

The following examples illustrate in detail the compositions of the instant invention and the method of using the same:

Example I

Using a xylene solution containing 5% pentachlorophenol and 4% of the amide of 3-dimethylaminopropylamine and acid A, maintained at a temperature of 25° C., test pieces of fir (2 inches x 4 inches x ¼ inch) are dipped in the solution for thirty seconds, and removed and air dried. Observations for blooming were made from twenty-four hours up to ten days, and it was noted that no blooming occurred.

Example II

Using xylene as the solvent, containing 5% pentachlorophenol and 5% of the amide of 3-diethylaminopropylamine and acid A, maintained at a temperature of 25° C., test pieces of fir (2 inches x 4 inches x ¼ inch) are dipped in the solution for 30 seconds, and removed and dried. Observations for blooming were made from twenty-four hours up to ten days, and it was noted that no blooming occurred.

In separate test runs the same procedure as that just described was carried out except that the acids used in forming the amide were acids B, C, D and F, and it was again noted that no blooming took place during the observation period.

Example III

Procedures were carried out that are the same as that described in connection with Example I, except that mineral spirits was used as the solvent and the concentrations of the amido compound were 4% and 5% and the amido compound used was a salt of 1 mol of acid A with 1 mol of an amide of 3-dimethylaminopropylamine and acid A; and no blooming took place during the observation period.

Example IV

A procedure was carried out that is the same as that described in the first paragraph of Example II except that the salt of 1 mol of acid A with the amide was used; and it was noted that no blooming took place during the observation period.

Example V

Using a xylol solution containing 5% pentachlorophenol and 5% of the salt of 1 mol of acid E with 1 mol of the amide 3-dimethylaminopropylamine and acid E, maintained at a temperature of 25° C., test pieces of fir are dipped in the solution for 30 seconds, and removed and dried. Observations for blooming were made from twenty-four hours up to ten days, and it was noted that no blooming occurred.

A procedure that is the same as that described in the foregoing paragraph, except that the salt of 1 mol of acid E with 1 mol of the amide of 3-diethylaminopropylamine and acid E was used, and the results obtained were the same.

Example VI

Using a xylene solution containing 5% pentachlorophenol and 4% of an amide of 3-diethylaminopropylamine and acid F, maintained at a temperature of 25° C., test pieces of fir are dipped in the solution for 30 seconds, and removed and dried. Observations for blooming were made from twenty-four hours up to ten days, and it was noted that no blooming occurred.

Substantially the same results are obtained using the procedure of the foregoing paragraph, except that the acid is acid A or acid D.

Example VII

Using a mineral spirits solution containing 5% pentachlorophenol and 5% of the salt of 1 mol of acid D with 1 mol of the amide of 3-dimethylaminopropylamine and acid D, maintained at a temperature of 25° C., test pieces of fir are dipped in the solution for 30 seconds, and removed and dried.

Observations for blooming were made from twenty-four hours up to ten days, and it was noted that no blooming occurred.

Example VIII

Using a mineral spirits solution containing 5% pentachlorophenol and 5% of the salt of 1 mol of acid F with 1 mol of the amide of diethylaminoethylamine and acid F, maintained at a temperature of 25° C., test pieces of fir are dipped in the solution for 30 seconds, and removed and dried. Observations for blooming were made from twenty-four hours up to ten days, and it was noted that no blooming occurred.

The same results are obtained using the procedure of the foregoing paragraph except that the acid used is acid B, C, D or A.

The same results are also obtained using the procedure of the first paragraph of this example except that only a 4% solution of amides of diethylaminoethylamine and acids F or B are used.

Example IX

A procedure is carried out that is the same as that described in the first paragraph of Example I except that mineral spirits is the solvent and a salt of ⅔ of a mol of acid A with the amide is employed, and the results obtained are the same.

Comparable results are obtained using each of the various other amides or salts thereof hereinbefore described. As mentioned previously, the particular manner in which the pentachlorophenol and the amido compound cooperate is not fully understood, although it is believed to involve some type of chemical association. In any event, blooming is substantially eliminated by the use of the instant amido compounds. In the case of other woods, such as pine, for example, the quantities of anti-blooming agent used may be slightly smaller than those used with fir, but within the range of proportions herein set forth the improvement of the instant invention is obtained.

Certain anti-blooming agents have met with commercial acceptance. The anti-blooming agents in current use, however, are associated with one or more of such disadvantages as poor solubility in solvents normally used to prepare the finished treating solution, a tendency to oxidize or undergo other chemical or physical changes so as to produce an insoluble residue in the treating solution either on storage or in use, a tendency to undergo chemical changes to produce or to accelerate the production of undesirable color in the treating solution (which color may stain the wood making it unsatisfactory for many uses), and a tendency to decrease the very desirable water repellency imparted to the wood through treatment or impregnation of the wood with the finished treating solution. The instant anti-blooming agents are unique in that they do not suffer from any of the aforementioned disadvantages.

It will also be appreciated that other additives ordinarily employed in the treatment of wood with pentachloro-

phenol may also be employed without subtracting from the results obtained in the practice of the instant invention.

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 composition comprising pentachlorophenol and an amido compound, dissolved in a common solvent, the concentration of the pentachlorophenol being 1% to 30% and the concentration of the amido compound being ½ to 10 times the pentachlorophenol concentration, said amido compound being selected from the group consisting of compounds having the formulae

$$RCO—NR'—A—NR_2''$$

and

$$RCO—NR'—A—NR_2''.HO—OCR$$

wherein RCO is an acyl group derived from a carboxylic acid selected from C_{12}–C_{20} fatty acids and rosin acids, A is a C_2–C_6 alkylene group, R′ is a radical selected from the group consisting of H and C_1–C_4 alkyl radicals, and R″ is a C_1–C_4 alkyl radical.

2. A composition comprising pentachlorophenol and N-dimethylaminopropyleneamide of rosin acid, dissolved in a common solvent, the concentration of the pentachlorophenol being 1% to 30% and the concentration of the N-dimethylaminopropyleneamide of rosin acid being ½ to 10 times the pentachlorophenol concentration.

3. A composition comprising pentachlorophenol and N-dimethylaminopropyleneamide of stearic acid, dissolved in a common solvent, the concentration of the pentachlorophenol being 1% to 30% and the concentration of the N-dimethylaminopropyleneamide of stearic acid being ½ to 10 times the pentachlorophenol concentration.

4. A wood preserving process which comprises contacting the wood with a composition comprising pentachlorophenol and an amido compound, dissolved in a common solvent, the concentration of the pentachlorophenol being 1% to 30% and the concentration of the amido compound being ½ to 10 times the pentachlorophenol concentration, said amido compound being selected from the group consisting of compounds having the formulae

$$RCO—NR'—A—NR_2''$$

and

$$RCO—NR'—A—NR_2''.HO—OCR$$

wherein RCO is an acyl group derived from a carboxylic acid selected from C_{12}–C_{20} fatty acids and rosin acids, A is a C_2–C_6 alkylene group, R′ is a radical selected from the group consisting of H and C_1–C_4 alkyl radicals, and R″ is a C_1–C_4 alkyl radical; and then drying the wood.

5. A wood preserving process which comprises contacting the wood with a composition comprising pentachlorophenol and N-dimethylaminopropyleneamide of rosin acid, dissolved in a common solvent, the concentration of the pentachlorophenol being 1% to 30% and the concentration of the N-dimethylaminopropyleneamide of rosin acid being ½ to 10 times the pentachlorophenol concentration; and then drying the wood.

6. A wood preserving process which comprises contacting the wood with a composition comprising pentachlorophenol and N-dimethylaminopropyleneamide of stearic acid, dissolved in a common solvent, the concentration of the pentachlorophenol being 1% to 30% and the concentration of the N-dimethylaminopropyleneamide of stearic acid being ½ to 10 times the pentachlorophenol concentration; and then drying the wood.

7. A composition comprising pentachlorophenol and an amido compound, dissolved in a common solvent, the concentration of the pentachlorophenol being 5% and the concentration of the amido compound being substantially equal to the pentachlorophenol concentration, said amido compound being selected from the group consisting of compounds having the formulae

$$RCO—NR'—A—NR_2''$$

and

$$RCO—NR'—A—NR_2''.HO—OCR$$

wherein RCO is an acyl group derived from a carboxylic acid selected from C_{12}–C_{20} fatty acids and rosin acids, A is a C_2–C_6 alkylene group, R′ is a radical selected from the group consisting of H and C_1–C_4 alkyl radicals, and R″ is a C_1–C_4 alkyl radical.

8. A wood preserving process which comprises contacting the wood with a composition comprising pentachlorophenol and an amido compound, dissolved in a common solvent, the concentration of the pentachlorophenol being 5% and the concentration of the amido compound being substantially equal to the pentachlorophenol concentration, said amido compound being selected from the group consisting of compounds having the formulae

$$RCO—NR'—A—NR_2''$$

and

$$RCO—NR'—A—NR_2''.HO—OCR$$

wherein RCO is an acyl group derived from a carboxylic acid selected from C_{12}–C_{20} fatty acids and rosin acids, A is a C_2–C_6 alkylene group, R′ is a radical selected from the group consisting of H and C_1–C_4 alkyl radicals, and R″ is a C_1–C_4 alkyl radical; and then drying the wood.

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