PATENT SPECIFICATION

DRAWINGS ATTACHED

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COMPLETE SPECIFICATION

Improvements in or relating to a Process for the Preservation of Wood

We, KOPPERS COMPANY, INC., a corporation organized under the laws of the State of Delaware, one of the United States of America, of 436 Seventh Avenue, City of Pittsburgh, Zone 19, Commonwealth of Pennsylvania, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the preservation of wood and more particularly is concerned with a process for impregnating wood with a preservative impregnating agent.

It is a common practice to treat wood chemically to increase its service life when the wood is to be used under conditions that favor the early deterioration of the wood, as by decay, insects, and other destructive agencies. This treatment of wood is generally carried out by forcing preservative chemicals into the wood by pressure. Conveniently this treatment involves subjecting the wood to a predetermined air pressure (super-atmospheric or sub-atmospheric) and, without releasing this pressure, introducing a bath of a preservative onto the wood, then increasing the pressure and usually elevating the temperature of the preservative nearly to the boiling point of water and maintaining this elevated pressure for an interval of time to obtain the desired injection of preservative in the wood. Thereafter, the bath of preservative is removed from the wood and the wood subjected to sub-atmospheric pressure, whereby the solvent evaporates from the wood while the preservative remains in the wood. The wood is now ready for use.

The treatment of the wood is conventionally denoted as a "full cell" or an "empty cell" process, depending upon the extent of retention of the preservative in the wood. These two processes differ mainly in the initial treating steps. The wood is subjected to sub-atmospheric pressure in the full cell process so as to empty the cells of air before the wood is subjected to the bath of preservative; and the wood is placed under super-atmospheric air pressure in the empty cell process to force a considerable amount of air into the cells of the wood before subjecting the wood to a bath of preservative so that when the pressure is released, the air in the cells will eject some of the preservative from the cells.

Conventionally an aqueous or an oil solution of a preservative is used as the bath. The aqueous or oil solvent, of course, has no preservative action but merely places the preservative in a form that will penetrate the cells of the wood. The presence of a great amount of water is, in fact, undesirable after the preservative is in the wood; thus, the use of water for many end uses requires the wood to be dried after impregnation of the wood with the preservative. This drying requires heat and time and is somewhat difficult, as the wood cell, in nature, normally includes water. The moisture content of thoroughly seasoned wood is, for example, within the range of six to fifteen per cent of the dry weight of the wood. Also the wetting and drying tends to cause checking of the wood and undesirable raising of the grain of the wood.

In normal treatments of wood to provide protection against fungus or termite attack; it is customary to use as an impregnating solution mixtures of liquid aromatic hydrocarbons, such as cresote, mixtures of inorganic salts which are dissolved in water, or certain solid organic compounds which are dissolved in petroleum distillates.

[Price 4s. 6d.]

Each of these types of impregnating solutions has certain inherent disadvantages. Liquid aromatic hydrocarbons impart to the wood strong odors and leave the wood with a surface which is for practical purposes very oily and difficult to paint. Wood treated with salts dissolved in water is expanded dimensionally due to the interaction of the water with the cellulose of the wood. This is partially overcome by kiln drying or prolonged air seasoning after treatment, but such drying results in degrade due to distortion, grain raising, and the like. Wood treated with organic preservatives dissolved in petroleum distillates have the same disadvantages as wood treated with the aromatic hydrocarbons. Using lower boiling petroleum distillates, such as mineral spirits, as the solvent, fails to eliminate the disadvantages completely. Several months of air seasoning after treatment are frequently required to permit sufficient evaporation of the solvent if the wood is to be painted. During this period of air reasoning a portion of the preservative can move to the surface of the wood with the solvent, thus the retention of preservative in the wood is reduced below that contemplated by (calculated during) the treatment. If the wood is shipped from the treating plant without air seasoning, it is necessary that each layer be separated from adjacent layers, otherwise sweating of the solvent between the layers occurs and the wood would be received at its destination with oil surfaces.

Many years ago, it was proposed to overcome the disadvantages of using the abovementioned solutions by employing as a preservative carrier dimethyl ether, a normally gaseous medium that is liquefied at ambient temperature under pressures above atmospheric. A full scale process was proposed and this led to high preservative injections. A major disadvantage of this process was the fact that the vapour pressure of dimethyl ether increased very rapidly with small increases in temperature, and if sufficient temperature was used to store enough heat in the wood to evaporate the dimethyl ether injected, then cylinder pressures above those recommended and permitted by specifications, would be developed. In addition, water contained in wood has appreciable solubility in dimethyl ether and such water that would dissolve in the solvent could not be separated without fractionation. This would strip the preservative out of the solvent, creating a considerable loss.

It is an object of this invention to overcome the disadvantages of using solutions as mentioned above. Wood processed in accordance with this invention is removed from the cylinder dimentionally unchanged, odourless and with non-oily surfaces. There is no subsequent loss of preservative due to solvent movement towards the surface. Air storage or kiln drying is not necessary.

Thus, according to one aspect of the present invention there is provided a process for the preservation of wood by impregnating the same with a preservative, which process includes the step of immersing the wood in a solution of a preservative impregnating agent in a liquefied aliphatic hydrocarbon and a cosolvent in which said preservative impregnating agent is soluble and with which the liquefied aliphatic hydrocarbon is readily miscible, said solution being maintained at elevated pressure and operating temperature and said liquefied aliphatic hydrocarbon boiling below the boiling point of water and being gaseous at the operating temperature and atmospheric pressure but liquid at the operating temperature and elevated pressure.

The invention provides a process which is particularly suitable for carrying out the impregnation of wood with preservative impregnants such as pentrachlorophenol. It has now been found that wood may be readily impregnated with a preservative when the preservative is in solution in an aliphatic hydrocarbon and a cosolvent therefor.

Thus, the invention also provides a wood preservative impregnating solution comprising a solution at elevated pressure of a preservative in a liquefied aliphatic hydrocarbon and a cosolvent to assist said preservative into solution, said aliphatic hydrocarbon being such that (a) it boils below the boiling point of water at ambient atmospheric pressure and (b) it readily liquefies at ambient temperature when placed under elevated pressure.

The preferred aliphatic hydrocarbons used in the preparation of the impregnating solution are propane, *n*-butane, iso-butane, *n*-pentane and iso-pentane and mixtures thereof such as petroleum gas; especially preferred are the butanes, propane or mixtures thereof.

These solvents are selected because of their desirable vapour pressure characteristics, availability and economy. In particular they can be heated to a temperature, without exceeding the maximum pressure limits of treating specifications, such that the heat contained in the solvents together with the hear transferred to the wood is sufficient to cause complete evaporation of this solvent containing in the wood at the conclusion of the treating cycle.

A cosolvent, miscible with the lower aliphatic hydrocarbon, it utilised to introduce the impregnant into the solvent medium. Methyl alcohol can be successfully used as a cosolvent for dissolving pentachloro-phenol in liquefied petroleum gases, in propane or in a blend of butane and propane. Other cosolvents suitble for introducing pentachlorophenol or other preservatives into the solvent vehicle for the intended purposes are toluene, acetone, benzene and various polyhydric alcohols.

The solvents for the wood impregnants are mixed in a manner such that they can be heated to a temperature sufficient to store enough heat within themselves and within the

wood, without exceeding 200° F. and without developing vapour pressures in excess of 200 lbs per square inch, so that the amount of solvent injected during the processing will be essentially self-evaporating when a vacuum is drawn on the cylinder containing the treated wood.

When solvents which have a relatively high boiling point at atmospheric pressure are used, it is desirable to use an empty cell process for treating, so as to reduce the injection and create a kick-back of the preservative at the conclusion of the pressure phase, thus reducing the quantity of solvent which must be self-evaporating. The non-soluble, non-condensable gas used in such empty cell process may be air, but preferably nitrogen or other non-combustible gas, due to the burnable characteristics of most solvents used. Such empty cell process may also be employed for treating wood even when the vapour pressure characteristic of the solvent used lends itself to a full cell process.

In the process of this invention the wood is subjected to a predetermined pressure and then, without changing pressure, subjected to solution of a preservative in a lower aliphatic hydrocarbon, the pressure is increased to inject the hydrocarbon into the wood, and thereafter the hydrocarbon evaporates from the wood.

The novel process of this invention is applicable to all commercial species of wood. In fact, it has been found that the process of this invention permits the impregnation of refractory species (those species of wood which tend to resist penetration) better than the conventional procedures.

Preservatives which may be used in the process of the invention include copper naphthenate, tetrachlorophenol, pentachlorophenol, *o*-chlorophenol, *β*-naphthol and chlorinated *β*-naphthol.

Water repellents such as paraffins, silicones and linseed oil as well as dyes are likewise employable for their intended purposes with these liquefied vehicles acting as a carrier for the water repellents.

The pressure necessary for treatment of wood is readily obtained by heating the treating solution while it is in the treating cylinder impregnating the wood therein, when such solvents as propane, butane, and iso-butane, are used. In this manner, the actual vapor pressure of the solution is increased sufficiently to give the required pressure for the impregnation of the wood. Resort to pressure applied from external sources, as by either a pressure pump or gas compressors, is necessary when solvent mediums, such as pentane or iso-pentane, are used, or when high volumetric injections are desired.

Because of the very low viscosity of the liquefied lower aliphatic hydrocarbons, the preservative solution penetrates refractory species, such as Douglas fir, much more readily than does any other preservative. Because the solvent is evaporated easily while the wood is still in the treating cylinder, "blooming" does not occur in service, and so, the treatment of the wood is more durable than other light solvent formulations.

The use of a cosolvent enables additional materials to be impregnated into wood the solubility of the impregnant to be increased so that a satisfactorily higher retention of a preservative can be injected into the wood. For example, pentachlorophenol has a relatively low solubility in liquefied petroleum gases *per se* but a solution containing a sufficient quantity of pentachlorophenol can be obtained by adding a cosolvent such as methyl alcohol or toluene, to the liquefied petroleum gases.

Water repellent chemicals soluble in the cosolvent mixture may also be added to the formulations, for example wax or paraffin.

The treatment of wood in accordance with this invention, wherein wood is impregnated with a preservative in a low boiling aliphatic hydrocarbon, has manifold advantages. The treated wood is clean, the increase in weight of the wood because of the treatment is due substantially to the preservative that is retained in the wood; and there is little, if any, blooming and subsequent loss of preservative due to solvent movement toward the surface. Also the solvent readily leaves the wood. Since the lower aliphatic hydrocarbons have no affinity for wood, as does water, and since the vapor pressures of the hydrocarbons are greater than the final pressure in the treating chamber, the hydrocarbons readily escape from the wood. Thus, air storage or kiln drying is not required. In fact, the hydrocarbon itself serves as a stripper for removing some of the residual free water, if present, from the wood, i.e., at the conclusion of the pressure phase, the rapidly expanding solvent gases expel a portion of the free water from the wood. The wood treated in accordance with this invention leaves the preservative bath substantially dimensionally unchanged. Wood treated in accordance with this invention is odorless and has a non-oily surface, an advantage, of course, if the wood is to be painted.

The treatment can be accomplished within a minimum of time. The extremely low viscosity of LPG, and the fact that no length pre-or post-treating processing is required, lowers the cost by increasing the throughput.

The fact that the carrier LPG (liquefied petroleum gas) may be recovered and re-used, lowers the cost.

While pentachlorophenol and copper naphthenate preservatives have been mentioned, any useful materials soluble in the lower aliphatic hydrocarbons and cosolvents, may be utilized.

Either a full cell or an empty cell process may be used for impregnating the wood, but it is generally desirable to use an empty cell

process as some solvent is removed from the wood by the expansion of the non-condensable gases, thus less heat is required to carrying out the volatilisation of the remaining solvent. However, with the use of an inert gas and proper manipulations of pressures with empty cell process, the retention of LP gas in the wood is reduced, and thus complete evaporation of the remaining LP gas in the wood is effected by the heat stored in the wood.

Compressed air may be used as the non-condensable gas, but because of the generally combustible nature of the solvents, it is preferable to use an inert gas, such as nitrogen. The inert gas or nitrogen is introduced into the treating cylinder before the treating solution is charged to the cylinder. In this manner, a cushion of inert gas is formed within the wood under the LPG treating solution. When the injection phase of the treating cycle is over, the compressed inert gas expands in the cells and forces a portion of the treating solution out of the wood. The quantity of solvent retained in the wood is thus lessened, and the time for LPG recovery is also thus shorter. This empty cell method also permits the operator to vary the net retention of the toxic without changing the concentration of the toxic in the LPG. Temperatures and pressures suitable for the process, range up to 200° F., and corresponding pressures ranging up to 200 pounds per square inch. Otherwise, the only limitation on temperatures and pressures suitable for the process, are those limitations as restricted by standard wood treating specifications, such as those included in Federal Specification TT-W-571c.

Blends of the liquids, such as a 50/50 mixture of propane and butane, provide a higher vapor pressure than straight butane, thus enabling the entrapped gas to escape quicker, even when less heat is applied during treatment.

More specifically, the present invention comprises the following series of steps as the best mode for commercially carrying out the main invention in a simple, economical, and practical way:

For full cell operation, the material to be impregnated is enclosed in a hermetically sealed cylinder. The air in the cylinder is evacuated. A solution containing the solvent, plus co-solvent, plus preservative, plus water repellent, if used, is introduced into the cylinder by first equalizing the pressure of the LP gas, etc., solution in a pressure storage tank with the vacuum in the treating cylinder. After the pressure is equalized, the cylinder is filled by pumping in additional preservative solution. The pressure is then raised by means of heat applied to the solution until the wood or other material is impregnated with the LPG, etc. Additional solution may be pumped into the cylinder, if necessary, to raise the pressure to desirable minimums.

When sufficient impregnant has been put into the wood, the liquid is returned from the cylinder through a cooler to the pressure storage tank, utilizing the vapor pressure developed in the cylinder to effect the transfer. A vapor pump is used to remove the vapors in the cylinder. These are passed through the same cooler or through a separate heat exchanger, cooled and condensed, then transferred to the storage tank. As the vapor pressure in the cylinder is reduced, solvent contained in the wood evaporates and these vapors are returned similarly to the storage tank. After the gases contained in the treated wood have been evaporated, condensed, and placed in the pressure storage tank, the treating cylinder is opened, and the wood withdrawn.

For empty cell operation, using an incombustible gas, the wood is enclosed in a hermetically sealed cylinder, the air evacuated, and an inert gas, such as nitrogen, is introduced at a relatively low pressure, such as 50 pounds per square inch, and forced into the wood. On top of this gas, the LPG, etc., is impregnated into the wood, resulting in a cushion of inert gas under pressure within the wood and under the LPG, etc. When the impregnating cycle is ended and the liquid LPG, etc. is returned to the pressure storage tank, there is little LPG, etc. in the gaseous state.

The quantity of LPG, etc. retained in the wood after treatment, is lessened and the cylinder can be opened earlier. This empty cell method of treatment also allows the operator to vary the net retention of the toxic, etc., without changing the concentration of the toxic in the LPG.

In addition to the general objects recited above, the invention has for other objects, such other improvements and advantages as may be found to obtain in the method and apparatus hereinafter described and claimed.

In the accompanying drawing forming a part of this specification, there is shown, for purposes of illustration, a flow diagram of the best mode of embodying and practicing the invention with pentachlorophenol.

Referring to the drawings:

PREPARATION OF TREATING SOLUTION:

To provide the amount of pentachlorophenol that will be required in liquefied petroleum gases (LPG) for the treating solution, methanol is used as the cosolvent. Pentachlorophenol (penta) is received in drums (not shown) and dumped at 10 into the penta mix pot 11. Methanol is also received in drums 12 which are connected by piping 13 to the liquid pump 14. Using this pump 14, the methanol is pumped from drums 12 through lines 13, 16, and the bypass line 17 around the penta mix pot 11 to the methanol-penta mix tank 15. When a sufficient amount of methanol has been charged from drums 12 to the mix tank 15, the

valves 18 on line 13 between the methanol drums 12 and the pump 14, are closed. The valve 19 in line 20 between the mix tank 15 and the pump 14, is then opened, valve 21 in line 17 is closed, valves 22 and 23 in lines 24 and 25 are opened, and the methanol is circulated by line 20 from the mix tank 15 to the pump 14, and back to the mix tank 15 through lines 16 and 24 to the penta mix pot 11 and line 25 to the mix tank 15. The penta crystals in tank 11 are dissolved in this manner in the methanol from tank 15 until a 50 per cent solution of penta in methanol is prepared. Pump 14 is then shut off and the valves 22, 21, 19 in line 24, 17 and 20, are closed. The 50 per cent solution of methanol-penta is then held in mix tank 15.

Liquefied petroleum gas (LPG) is received at 26 in tank trucks and unloaded with the truck's pump into the LPG storage tank 27 and the LPG-penta storage tank 28. The final step in the preparation of the treating solution is the addition of methanol-penta solution from the mix tank 15 to the LPG from tanks 27, 28, to prepare a solution containing the necessary percentages of methanol, penta, and LPG or solvent vehicle or formulation. A representative example for full cell operation would be 4 per cent methanol, 4 per cent penta, and 92 per cent LPG. A representative example for empty cell operation would be 6 per cent methanol, 6 per cent penta and 88 per cent formulation. This is accomplished by introducing an inert gas, such as nitrogen, from line 29 to the mix tank 15, to increase its pressure to a point equal to the pressure in storage tank 28. Then valve 30 in line 21, and 32 in line 33, valve 36 in line 37, and valve 34 in line 35, are opened, and pump 14, is started. This will circulate the LPG from the tank 28 to pump 14 and back through lines 35, 37, to tank 28. Next, the methanol-penta mix from mix tank 15 is added into the suction side of pump 14 while the pump is circulating LPG tank 28. To do this, the valve 19 in mix tank line 20 to the pump 14 is opened slightly. When the required amount of methanol-penta is added from mix tank 15, the valve 19 is closed. The liquid pump 14 continues to circulate the mix through tank 28 until a uniform mixture of LPG-penta solution is obtained, whereupon pump 14 is stopped, and valves 34, 36, 30 and 32 are closed.

A typical treating solution might be formulated, using 92 per cent butane, 4 per cent pentachlorophenol, and 4 per cent methanol.

The concentration of the pentachlorophenol in the treating solution can be varied, depending upon the retention desired or specified.

FULL CELL TREATMENT:

The cylinder 38 is charged with wood and the cylinder door 39 is closed and sealed. All valves for the cylinder 38 are closed except the valve in line 40 between the cylinder 38 and the ejector 41. Next, the steam to the ejector 41 is turned on through line 42 and valve 43, and the cylinder 38 evacuated to 26-in. Hg. vacuum. This will remove air from the cylinder 38 and the wood therein. Treating solution can now be added to the cylinder. Valves 30 and 32 in lines 31 and 33 between the storage tank 28 and pump 14 are opened. Valves 44 in line 45 between line 16 from pump 14 and the treating cylinder 38, are opened. Pump 14 is started and the treating solution is charged to the cylinder 38. At the same time, the steam to the ejector 41 is shut off and the valve 40 in line 44 to the ejector, is closed. Treating solution is added to the cylinder 38 until the wood charge is covered with treating solution. All valves 30, 32, 44, between tank 28, pump 14 and the treating cylinder 38, are then closed. The next step is to increase the pressure in the cylinder to about 150 Psig. This is accomplished by turning steam from line 45 and valve 46 into the coils 47 of the treating cylinder 38. The temperature of the solution is increased to 180° F., at which temperature the solution has a vapor pressure of about 150 Psig. The pressure in the cylinder 38 is controlled automatically by a recording pressure controller 48 which controls the steam flow to the heating coils 47 in the cylinder 38. This pressure is maintained for sufficient time, such as, two to three hours, to effect the impregnation of the wood 48[1] with the treating solution.

At the conclusion of the pressure cycle the treating solution is transferred from the treating cylinder 38 to the LPG-penta hold tank 49 rather than being returned to the LPG-penta storage tank 28. This transfer is accomplished by opening valve 50 in line 51, between the cylinder 38 and the condenser cooler 52, and valve 53 in line 54, valve 55 in line 56, between the cooler 52 and the line 35 to the line 57 to the hold tank 49 as well as opening valve 58 in line 57. This transfer can be made without the use of the pump 14, since the pressure in the cylinder 38 will be about 100 Psig greater than the pressure in the hold tank 49. As the liquid treating solution passes to the hold tank 49, it is cooled in the condenser cooler 52. This cooling reduces the vapor pressure and thereby aids the transfer. Any LPG-penta treating solution remaining in the cylinder 38 at the conclusion of the liquid transfer will be transferred to the hold tank 49, using the liquid pump 14. At the conclusion of the liquid transfer, the steam line 45 to the cylinder 38 heating coils 47, is shut off. Considerable LPG remains in the cylinder 38, some as a liquid in the wood, and the remaining as vapor around the wood. This LPG is recovered, using vapor pump 59. This vapor pump removes the LPG vapor from the cylin-

der and compresses it to 150 psig. This is accomplished through opening valve 60 in the line 61. From the vapor pump 59, the compressed gas passes through line 62 and valve 63 to the condenser cooler 52. Here, the condensable gases are passed through line 54 and valve 64 to collecting receiver 65. Non-condensable gases are vented through line 66 pressure controller 67 to the atmosphere. The condensed material collected in receiver 65 is released to the hold tank 49 through the liquid level controller 68 in lines 56, 35, and 57. As the pressure in the cylinder 38 is reduced by the operation of the vapor pump 59, the liquid LPG impregnated in the wood begins to vaporize and come out of the wood. This is accelerated by the fact that the wood, warmed during treatment by coils 47, supplies the heat necessary to vaporize the LPG. When the vapor pump 59 no longer removes LPG in any quantity from the cylinder 38, the valves 60, 69 in line 61 and 62 are closed. Final traces of LPG in the cylinder are removed by ejector 41. The valve 40 in line 44 is opened and the steam in line 42 to the ejector 41, is turned on. A final vacuum of 26 in. of Hg. will be maintained on the cylinder 38 for about 1/2 hr. This will remove any remaining LPG. At the conclusion of the vacuum period, before the ejector 41 is turned off, the cylinder 38 is flushed with inert gas through lines 70, 71, from the inert gas receiver 72. This will completely flush out all final traces of LPG. The ejector 41 is then turned off and the vacuum is broken with inert gas from receiver 72. The cylinder door 39 is then opened and the wood removed.

The used treating solution, now in the LPG-penta hold tank 49, is now metered and analyzed. Penta content of this solution will be a check on the retention of the charge. Any water picked up in the treating cylinder or from the wood is decanted from the treating solution by way of the water drain valve 75 of the tank 49. The penta content is then adjusted to 4 per cent by the addition of methanol-penta solution from the methanol-penta mix tank 15. Valve 73 in line 74, and valve 32, in the line 33 between the hold tank 49 and the liquid pump 44, are opened. Valves 34, 58 in lines 35 and 57 between the liquid pump 14 and the hold tank 49, are also opened. Pump 14 is then started and the solution circulated. The valve 19 in line 20 is then opened and the required amount of alcohol-penta is added from mix tank 15 to the system. The valve 19 in line 20 is then closed. The liquid pump 14 will continue to circulate the hold tank 49 until a uniform solution is obtained. Then the valve 58 in line 57 is closed and the valve 36 in line 37 is opened. In this manner, the adjusted treating solution is transferred to the LPG-penta storage tank 28 and is now ready to treat the next charge.

EMPTY CELL TREATMENT:

The empty cell treatment is employed when it is desired to process wood in which a lower amount of preservative is to be retained in the wood and is identical to the full cell treatment except that after the initial vacuum the wood is pressured with inert gas at 20 to 30 psig. Other gas pressures are used also, depending upon the final results desired. Without relieving this pressure from tank 72, the treating solution from storage tank 28 is introduced into the cylinder 38. After this the treatment is identical with the full cell treatment above-described.

The following examples are some of the actual tests made in accordance with the foregoing described process procedure:

EXAMPLE I

Kiln dried timbers of southern pine were placed in a closed cylinder and a vacuum of 25 inches of mercury was maintained on the cylinder for one half hour. Then, without admitting air, the cylinder was filled with a saturated solution of pentachlorophenol in pentane and methanol. The cylinder was then heated until the pressure in the cylinder increased to 125—200 pounds per square inch gauge. During the heating period, additional solution was pumped into the cylinder to replace the material forced into the wood and to maintain the pressure at 125—200 pounds per square inch gauge pressure, and these pressures and temperatures were maintained on the wood for about two hours. Then, the hydrocarbon/alcohol solution was removed from the cylinder and the cylinder subjected to a vacuum 25 inches of mercury for an hour. Thereafter the wood was removed from the cylinder. The wood appeared substantially dry, was clean and odorless. The wood had a high degree of penetration of the material and had therein 0.4 pounds of pentachlorophenol per cubic foot.

EXAMPLE II

Kiln dried timbers of southern pine were placed in a closed container and air was injected into the container until the pressure in the container reached 50 pounds per square inch gauge. Thereafter, a saturated solution of pentachlorophenol in butane and methanol was forced into the container and air withdrawn from the container at a rate to maintain a constant pressure of 50 pounds per square inch gauge in the container. After the solution had filled the container, the pressure was increased to 200 pounds per square inch gauge by raising the temperature, this pressure was maintained for two hours and then released. As soon as this pressure was released, the compressed air in the wood expanded and forced the preservative from the wood to a considerable extent. The preservative was drained from the cylinder and the wood subjected to a

vacuum of 25 inches of mercury for a period of an hour, during which time the heat in the wood evaporated the solvent remaining. The wood now had therein about 0.2 pounds of pentachlorophenol per cubic foot.

EXAMPLE III

Green southern pine was placed in a closed container and subjected to steam at a pressure of 15 pounds per square inch gauge for six hours. The steam was released and the container evacuated for a period of two hours. Thereafter, a saturated solution of pentachlorophenol in butane and methanol was pumped into the container and, after the solution filled the container, the solution was heated to increase the pressure to about 200 pounds per square inch gauge and this pressure was then maintained for two hours. The pressure was then released, the preservative drained from the cylinder and the wood subjected to a vacuum of 25 pounds per square inch gauge mercury for a period of thirty minutes. The green wood presented a product that had been impregnated with preservative and partially dried.

EXAMPLE IV

Southern yellow pine samples were treated with LPG-penta solutions using the empty cell method. Treatment was carried out at 140—150 psig for approximately two (2) hours. These samples contained both sapwood and heartwood. There was no discernible swelling, warping, checking or distortion of the wood. The treated wood was clean and dry.

EXAMPLE V

1-in. by 6-in. by 24-in. panels of Southern yellow pine, sugar pine and Douglas fir were treated with a solution of 4 per cent by weight pentachlorophenol in butane and methanol. Full cell treatment was employed. The pressure for treatment was obtained by increasing the temperature in the treating cylinder to 130° F. Pressure used was 150 psig. All butane/methanol solvent mixture was removed from the wood in the treating cylinder. No apparent swelling, warping or grain raising was noticed. Penetrations were uniform.

EXAMPLE VI

3/4-in. by 3/4-in. by 40-in. Southern Yellow Pine field test saplings, and 2-in. by 4-in. by 48-in. samples of Douglas fir were treated in the same manner as Example V. Examination of the specimens showed uniform penetration with no apparent grain raising, swelling or checking.

EXAMPLE VII

A 1-in. by 1-in. by 9-in. piece of Southern yellow pine was treated by the empty cell method with a 5 per cent by weight pentachlorophenol in butane and methanol. Pressure of 162 psig held for 3 hours by heating the treating solution. When removed from cylinder the sample was clean and dry.

In operation the process and apparatus is manipulated in general in the same way as that described above with reference to the flow on the accompanying drawing.

WHAT WE CLAIM IS:—

1. A process for the preservation of wood by impregnating the same with a preservative which process includes the step of immersing the wood in a solution of a preservative impregnating agent in a liquefied aliphatic hydrocarbon and a cosolvent in which said preservative impregnating agent is soluble and with which the liquefied aliphatic hydrocarbon is readily miscible, said solution being maintained at elevated pressure and operating temperature and said liquefied aliphatic hydrocarbon boiling below the boiling point of water and being gaseous at the operating temperature and atmospheric pressure but liquid at the operating temperature and elevated pressure.

2. A process according to Claim 1, wherein the preservative impregnating agent is pentachlorophenol.

3. A process according to Claim 1 or 2, wherein the liquefied aliphatic hydrocarbon is petroleum gas, propane, *n*-butane, iso-butane, *n*-pentane, iso-pentane, neo-pentane or a mixture of two or more thereof.

4. A process according to Claim 1, 2 or 3, wherein the cosolvent is methanol.

5. A process according to any one of Claims 1 to 4, wherein the operating temperature does not exceed 200° F. and the elevated pressure does not exceed 200 psig.

6. A process according to any one of the preceding claims, which process includes the steps of (a) enclosing the wood in a hermetically sealable treating cylinder and evacuating air from the cylinder after the sealing thereof, (b) introducing an inert, incombustible, non-condensable gas at a pressure of up to 50 psig into the treating cylinder so as to inject the pores of the wood therewith, (c) without releasing said pressure, introducing said solution of a preservative impregnating agent into said treating cylinder so as to immerse the wood in said solution, (d) heating said solution with the wood immersed therein to the operating temperature whilst maintaining the solution in the liquid state under pressure, thereby increasing the pressure exerted on the solution to an extent such that some of the solution is forced into the pores of the wood immersed therein, (e) reducing the pressure exerted on said solution and recovering the solvents for the preservative impregnating agent from the wood and (f) purging the wood of residual solution.

7. A process according to any one of Claims 1 to 5, which process includes the steps of (a) enclosing the wood in a hermetically sealable treating cylinder and evacuating air from the

vacuum of 25 inches of mercury for a period of an hour, during which time the heat in the wood evaporated the solvent remaining. The wood now had therein about 0.2 pounds of pentachlorophenol per cubic foot.

EXAMPLE III

Green southern pine was placed in a closed container and subjected to steam at a pressure of 15 pounds per square inch gauge for six hours. The steam was released and the container evacuated for a period of two hours. Thereafter, a saturated solution of pentachlorophenol in butane and methanol was pumped into the container and, after the solution filled the container, the solution was heated to increase the pressure to about 200 pounds per square inch gauge and this pressure was then maintained for two hours. The pressure was then released, the preservative drained from the cylinder and the wood subjected to a vacuum of 25 pounds per square inch gauge mercury for a period of thirty minutes. The green wood presented a product that had been impregnated with preservative and partially dried.

EXAMPLE IV

Southern yellow pine samples were treated with LPG-penta solutions using the empty cell method. Treatment was carried out at 140—150 psig for approximately two (2) hours. These samples contained both sapwood and heartwood. There was no discernible swelling, warping, checking or distortion of the wood. The treated wood was clean and dry.

EXAMPLE V

1-in. by 6-in. by 24-in. panels of Southern yellow pine, sugar pine and Douglas fir were treated with a solution of 4 per cent by weight pentachlorophenol in butane and methanol. Full cell treatment was employed. The pressure for treatment was obtained by increasing the temperature in the treating cylinder to 130° F. Pressure used was 150 psig. All butane/methanol solvent mixture was removed from the wood in the treating cylinder. No apparent swelling, warping or grain raising was noticed. Penetrations were uniform.

EXAMPLE VI

3/4-in. by 3/4-in. by 40-in. Southern Yellow Pine field test saplings, and 2-in. by 4-in. by 48-in. samples of Douglas fir were treated in the same manner as Example V. Examination of the specimens showed uniform penetration with no apparent grain raising, swelling or checking.

EXAMPLE VII

A 1-in. by 1-in. by 9-in. piece of Southern yellow pine was treated by the empty cell method with a 5 per cent by weight pentachlorophenol in butane and methanol. Pressure of 162 psig held for 3 hours by heating the treating solution. When removed from cylinder the sample was clean and dry.

In operation the process and apparatus is manipulated in general in the same way as that described above with reference to the flow on the accompanying drawing.

WHAT WE CLAIM IS:—

1. A process for the preservation of wood by impregnating the same with a preservative which process includes the step of immersing the wood in a solution of a preservative impregnating agent in a liquefied aliphatic hydrocarbon and a cosolvent in which said preservative impregnating agent is soluble and with which the liquefied aliphatic hydrocarbon is readily miscible, said solution being maintained at elevated pressure and operating temperature and said liquefied aliphatic hydrocarbon boiling below the boiling point of water and being gaseous at the operating temperature and atmospheric pressure but liquid at the operating temperature and elevated pressure.

2. A process according to Claim 1, wherein the preservative impregnating agent is pentachlorophenol.

3. A process according to Claim 1 or 2, wherein the liquefied aliphatic hydrocarbon is petroleum gas, propane, *n*-butane, iso-butane, *n*-pentane, iso-pentane, neo-pentane or a mixture of two or more thereof.

4. A process according to Claim 1, 2 or 3, wherein the cosolvent is methanol.

5. A process according to any one of Claims 1 to 4, wherein the operating temperature does not exceed 200° F. and the elevated pressure does not exceed 200 psig.

6. A process according to any one of the preceding claims, which process includes the steps of (a) enclosing the wood in a hermetically sealable treating cylinder and evacuating air from the cylinder after the sealing thereof, (b) introducing an inert, incombustible, non-condensable gas at a pressure of up to 50 psig into the treating cylinder so as to inject the pores of the wood therewith, (c) without releasing said pressure, introducing said solution of a preservative impregnating agent into said treating cylinder so as to immerse the wood in said solution, (d) heating said solution with the wood immersed therein to the operating temperature whilst maintaining the solution in the liquid state under pressure, thereby increasing the pressure exerted on the solution to an extent such that some of the solution is forced into the pores of the wood immersed therein, (e) reducing the pressure exerted on said solution and recovering the solvents for the preservative impregnating agent from the wood and (f) purging the wood of residual solution.

7. A process according to any one of Claims 1 to 5, which process includes the steps of (a) enclosing the wood in a hermetically sealable treating cylinder and evacuating air from the

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cylinder after the sealing thereof, (b) equalizing the pressure in a pressure storage tank containing the solution of preservative impregnating agent with the vacuum in the treating cylinder, (c) introducing said solution into the treating cylinder until it is substantially full, (d) heating the solution to the operating temperature thereby raising the pressure in the treating cylinder and maintaining the raised pressure on the solution until the wood is impregnated therewith, (e) removing the solution from the treating cylinder and evacuating residual gases from the treating cylinder and wood therein so as to create a vacuum in the treating cylinder with the wood still therein and (f) thereafter flushing the cylinder with the wood therein with an inert gas.

8. A process for the preservation of wood by impregnating the same with a preservative, substantially as hereinbefore described with reference to the accompanying drawing.

9. Wood whenever treated by the process claimed in any one of the preceding claims.

10. A wood preservative impregnating solution comprising a solution at elevated pressure of a preservative in a liquefied aliphatic hydrocarbon and a cosolvent, with which the liquefied aliphatic hydrocarbon is readily miscible, to assist said preservative into solution, said aliphatic hydrocarbon being such that (a) it boils below the boiling point of water at ambient atmospheric pressure and (b) it readily liquefies at ambient atmospheric temperature when placed under elevated pressure.

11. A wood preservative impregnating solution as claimed in Claim 10, wherein the preservative is pentachlorophenol.

12. A wood preservative impregnating solution as claimed in Claim 10 or 11, wherein the aliphatic hydrocarbon is liquefied petroleum gas, propane, *n*-butane, iso-butane, *n*-pentane, iso-pentane or a mixture of two or more thereof.

13. A wood preservative solution as claimed in Claim 10, 11 or 12, wherein the cosolvent is methanol.

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