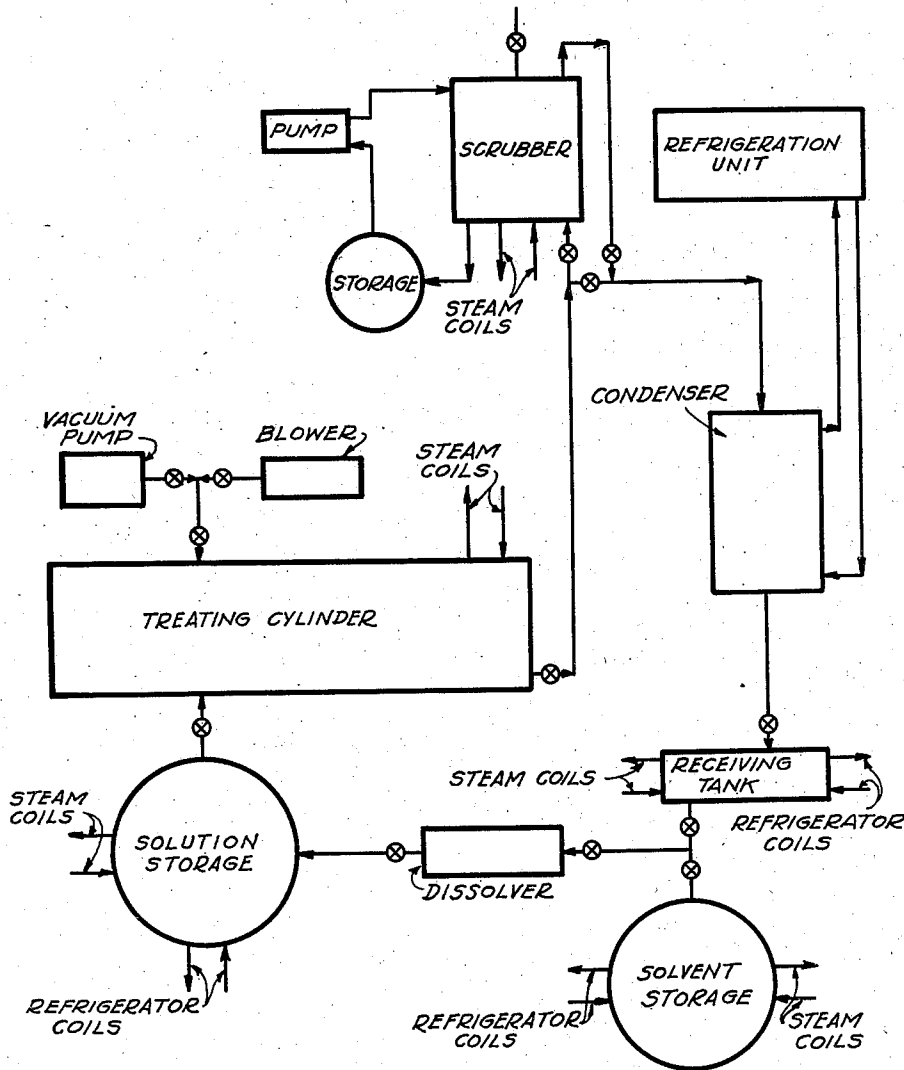


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IMPREGNATION OF POROUS MATERIALS

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IMPREGNATION OF POROUS MATERIALS

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This invention relates to processes for depositing preservative compositions, fireproofing compositions, and the like, within a porous material, and is particularly directed to processes where-
5 in such materials are carried into the porous materials in a fluid of very low boiling point.

In order to impregnate a porous material with suitable impregnating agents, it is now the customary practice to employ an aqueous menstruum. It is frequently disadvantageous to employ
10 an aqueous vehicle for impregnating agents because water has a deleterious influence upon certain porous materials. Cloth or rope, for instance, may be caused to shrink, and in some instances may be rather severely damaged. Another disadvantage arising from the use of aqueous
15 vehicles is the relatively long time required effectually to impregnate porous materials with water solutions of impregnating agents, and the even longer time ordinarily required satisfactorily
20 to dry the materials after impregnation.

The use of an aqueous menstruum for impregnating agents is particularly unsatisfactory when it is desired to impregnate wood. It is the customary practice, for instance, to preserve wood by
25 submerging it in a water solution of a preserving salt, such as zinc chloride, and then injecting the solution into the wood by the use of pressure. After a desired degree of penetration of the wood
30 has been effected, the aqueous solution is ordinarily allowed to drain from the pressure receptacle. Water which is retained in the wood must subsequently be removed by kiln drying or air seasoning.

The injection of water into the wood causes considerable swelling and, upon drying, the consequent shrinkage of the wood is attended by a certain amount of warping, checking, and raising
40 of the grain. The extent of this deterioration of the wood depends largely, of course, upon the characteristics of the wood being treated and, to a lesser degree, upon the care exercised in the treating and drying procedures. When the treated wood is to be employed in certain relations,
45 this deterioration is not particularly disadvantageous, but wood which is warped and checked cannot satisfactorily be used for a large number of purposes.

Aqueous vehicles are further unsatisfactory because of the relatively long time and the high pressures required to effect a satisfactory impregnation of wood. Moreover, the large amount of time required to dry the wood after its impregnation with an aqueous menstruum results in high
50 equipment and operating costs if it is kiln dried,

and in high carrying charges if it is air seasoned.

The customary processes of impregnating wood by means of an aqueous vehicle have also been found disadvantageous by reason of the relatively large and expensive equipment required for the ordinary pressure processes. In addition to the expense attendant upon the installation and maintenance of such equipment, there is the additional disadvantage that the equipment is not readily adaptable to various types of wood which
10 require modified methods of treatment.

Aqueous vehicles offer yet another disadvantage in that a large number of impregnating agents are not water soluble and cannot, therefore, be used in water solution.

Considered together, the above enumerated disadvantages have prevented the impregnation of wood by the use of an aqueous menstruum in many commercial applications. It has been impractical to impregnate finished or semi-finished
20 lumber with suitable agents for staining, fireproofing, or preserving the wood.

It has been proposed to avoid the difficulties encountered when porous materials are impregnated by the use of aqueous vehicles, by using
25 non-aqueous menstruums. It has, for instance, been suggested that alcohol, or liquid hydrocarbons such as gasoline or kerosene be used. Such non-aqueous vehicles display the same disadvantages as do aqueous vehicles, to a greater or
30 lesser extent, and they have the further disadvantage of being quite expensive. In addition to the fact that the non-aqueous solvents heretofore employed are high in cost for an initial installation, their expense is rendered even greater
35 by the fact that it is almost impossible to recover the solvent from the porous material.

When a porous material such as wood is to be impregnated with a preservative salt, such as zinc chloride, if a non-aqueous menstruum is to be
40 employed, it has been proposed to use alcoholic solutions of zinc chloride. Alcohol causes warping and checking of wood, though to a somewhat smaller extent than does water. Alcohol, moreover, causes a raising of the grain of the wood,
45 and, accordingly, like water, is unsuitable for treating finished wood.

After such a non-aqueous menstruum has been injected into wood, either by pressure processes
50 or by simple immersion, it is impractical to attempt recovery of the menstruum. It is, of course, theoretically possible to recover such non-aqueous menstruums, but the cost of such operations would be prohibitive. In a pressure process of
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impregnation, there is, of course, a certain amount of heat in the wood, but this heat is inadequate to distill off the relatively high-boiling menstrooms heretofore used. The poor heat conduction of wood, moreover, makes it impractical to distill off the vehicles by the application of heat from an external source. It is the ordinary practice to recover only the portion of the non-aqueous vehicle which can be drained from the wood, and no attempt is made to recover the fluid retained in the wood.

Processes employing non-aqueous menstrooms for the pressure impregnation of porous materials require equipment comparable in size and cost to that used in processes employing aqueous vehicles. Processes employing non-aqueous menstrooms also require relatively long periods of time for the impregnating and drying operations.

It is an object of my invention to provide processes which can be employed for the impregnation of porous materials without damage thereto. It is a further object of my invention to provide processes whereby wood may be impregnated with desired impregnating agents without the deleterious swelling, shrinking, warping, checking, and raising of the grain which attends the use of most of the menstrooms hitherto known. A still further object of my invention is to provide processes whereby finished lumber may satisfactorily be impregnated with suitable fireproofing, staining, and preservative agents. A still further object of my invention is to provide processes whereby a deep and uniform penetration of impregnating agents can quickly and economically be achieved. A still further object of my invention is to provide processes which are low in cost, and which can readily be adapted to the condition of the porous material. Other objects of my invention will become apparent hereinafter.

My objects are accomplished, briefly, by impregnating porous material with suitable impregnating agents carried in a non-aqueous menstruum which boils at a temperature not substantially higher than about 5° C.

The processes of my invention may advantageously be employed with a wide variety of porous materials, such as wood, textiles, rope, and the like. Such porous materials may be impregnated according to the processes of my invention with impregnating agents such as pigments, lakes, dyes, stains, resins, gums, lacs, oils, waxes, parasticides, and fireproofing compositions.

While the processes of my invention are applicable to porous materials generally, they are particularly advantageous for the impregnation of wood, and are especially so when it is desired to obtain a relatively great depth of penetration or when difficultly penetrable species are to be treated.

As has been above indicated, a wide variety of impregnating agents may be used according to my invention. Many water insoluble impregnating agents can be dissolved in the non-aqueous menstrooms of this invention, and it is thus possible to use such water insoluble impregnating agents in an economical and practical manner. For instance, such preservatives as beta-naphthol, tetrachlorophenol, and orthophenylphenol may be used in a suitable non-aqueous menstruum which boils not substantially above 5° C., such as dimethyl ether. I may also use mixtures of various impregnating agents.

According to the processes of my invention, I may employ any non-aqueous menstruum which boils at a temperature not substantially higher

than 5° C. I may use, for instance, such compounds as dimethyl ether, propane, butane, and methyl chloride. It will be understood that I may use mixtures of such compounds with each other or with higher boiling compounds, though in every instance it is preferred that the non-aqueous menstruum boil at a temperature not substantially higher than about 5° C. In addition to using low boiling liquids and low boiling mixtures of the type above discussed, I may also employ low boiling compounds in which a suitable lower boiling material has been dissolved. I may, for instance, use liquid dimethyl ether in which is dissolved as much as 20 to about 25 per cent carbon dioxide.

The above described menstrooms are solvents for a wide variety of impregnating agents, but there may be found an impregnating agent which is not soluble in a commercially available liquid which boils at a temperature not substantially higher than about 5° C. In such an instance, some of the advantages of my invention may be obtained by dispersing the impregnating agent in the menstruum, but it is usually desirable to employ impregnating agents which are soluble in the menstruum which is to be used.

The impregnation of porous materials may be accomplished by simply immersing them in the non-aqueous vehicles. When wood is treated, however, it is usually preferred to inject the non-aqueous vehicle thereto by the use of pressure. Pressures up to the critical pressures of the substances used as vehicles may be obtained by heating the liquid. As the non-aqueous liquids used boil at about 5° C. or below, no large amount of heat will be required to obtain relatively high pressures, and, under many circumstances, satisfactorily high pressures may be obtained without the addition of external heat by reason of the normal pressure of the liquids at ordinary temperatures.

The low boiling liquids which I employ are characterized, of course, by relatively low surface tensions and relatively low viscosities, and the impregnation of wood according to the processes of my invention proceeds very rapidly even at rather moderate pressures, a satisfactory degree of penetration being obtained in a relatively short period of time. This, of course, is very important from an economic standpoint because smaller equipment may be used for handling a given amount of lumber.

After a desired degree of penetration has been obtained, the liquid menstruum is drained from the wood. After the liquid has been drained off, the liquid remaining in the wood can readily be removed therefrom by permitting it to boil off. Ordinarily, no addition of heat is necessary to effect an almost complete evaporation of the liquid menstruum, but, if desired, additional heat may be supplied. The gas which results from the boiling of the liquid menstruum can be recovered and condensed, whereupon it may be used as a solvent for more of the impregnating agent.

It will be apparent that the removal of the non-aqueous low boiling menstrooms of my invention is exceedingly easy of accomplishment, and it is to be noted that a substantially complete recovery of the menstrooms is practical. The short time required to effect a separation of the liquid menstrooms from the wood is highly advantageous, of course.

In order that the preferred sequence of steps in a specific process for the impregnation of wood may be better understood, there is illustrated in

the accompanying drawing a flow sheet of such a typical process.

Wood to be impregnated is placed in the treating cylinder, and a solution of a suitable impregnating agent in a non-aqueous menstruum which boils at a temperature not substantially higher than 5° C. is admitted to the treating cylinder from the solution storage tank. The wood to be impregnated is placed in the treating cylinder and subjected to a vacuum, by means of the indicated vacuum pump, to remove most of the air from the cylinder and the wood. The cylinder is then connected to the solution storage tank, which is at a higher temperature than the cylinder, and the solution of impregnating agent flows into the cylinder under its own pressure.

The desired temperatures and the corresponding pressures are maintained in the treating cylinder by the use of steam coils. The desired temperature and pressure are maintained for the time required to effect the desired degree of penetration of the wood. At the end of this time, the line between the treating cylinder and the solution storage tank is opened, and the solution is forced from the treating cylinder into the solution storage tank which at this time, of course, is at a lower temperature and pressure than the treating cylinder.

After the solution has been drained from the wood and forced into the solution storage tank, the line between the cylinder and the solution storage tank is closed, and the line from the treating cylinder to the condenser is opened. The menstruum which has been retained in the wood distills therefrom and is condensed in the condenser from which it is led to a receiving tank. The temperature of the wood is ordinarily sufficient to supply the heat required to vaporize substantially all of the menstruum.

The atmosphere of the vaporized menstruum which remains in the treating cylinder is finally swept out with air from a blower, and dissolved in a suitable solvent in a scrubber. From time to time the menstruum which has been dissolved in the scrubber is removed from the dissolving liquid by heating, and the vaporized menstruum is returned through the condenser to the receiving tank and then to the solution storage tank.

The condensed liquid in the receiving tank is heated somewhat and forced, under its own pressure, through the dissolver where a desired amount of the impregnating agent is put into solution. It will be noted that a suitable storage tank for the liquid menstruum is provided, which storage tank is equipped with heating and cooling means whereby the pressure may be adjusted.

After the liquid menstruum has been evaporated from the wood and any remaining gases have been swept from the cylinder by means of the blower, the wood, which is now entirely dry, can be removed from the cylinder. The wood is then ready for use, and no further seasoning or drying is required.

Considering my invention with more particular reference to certain illustrative impregnating agents and certain non-aqueous menstrooms which have a boiling point not substantially higher than about 5° C., the following examples are given:

Example I

Following the procedure above outlined, a number of samples of finished wood were subjected to a 5% solution of zinc chloride in dimethyl ether. Temperatures of from 40 to 50°

C. were maintained with corresponding pressures from 130 to 150 pounds per square inch. These conditions were maintained for 30 minutes. The woods thus treated were as follows: California white pine, heart and sap, 1½" x 2" x 4"; Wisconsin white pine, sap, ¾" x 2" x 4"; Douglas fir, heart, 1¼" x 1¼" x 4"; red oak, heart, ¾" x 4" x 4". All of these specimens treated according to the procedure of this example were completely impregnated with zinc chloride. There was no discernible swelling, warping, or raising of the grain of these finished pieces of wood.

Example II

A cylinder of seasoned heart white oak, 3½" in diameter and 4" long, was subjected to a 5% solution of zinc chloride in dimethyl ether for three hours at temperatures of 40 to 50° C., and at corresponding pressures of 130 to 150 pounds per square inch. The wood used in this example is practically impenetrable by aqueous solutions, but under the conditions of this example, approximately half of the wood was impregnated with zinc chloride. There was no apparent swelling, checking, or distortion of the wood.

Example III

Following the procedure of the above examples, a number of pieces of ponderosa pine window sash were treated with a 5% solution of zinc chloride in dimethyl ether. The treatment was conducted at 150 pounds per square inch pressure for thirty minutes for sapwood and forty-five minutes for heartwood. There was no perceptible swelling, warping, checking, or roughening of the surfaces of the wood. Examination of typical specimens showed complete penetration of the wood with zinc chloride, and about one pound of zinc chloride was retained per cubic foot.

Example IV

Following the procedure of Example III, but using dimethyl ether containing dissolved therein about twenty per cent of carbon dioxide as the solvent for zinc chloride, a number of pieces of finished lumber were impregnated. Excellent results were obtained, and it is noted that a somewhat more rapid penetration of the wood seemed to be obtained than when the dimethyl ether was used alone as a solvent.

Example V

Wood was impregnated with a 2% solution of copper naphthenate in a commercial mixture of propane and butane. The mixture, which contained only a small amount of butane, boiled at -36° C. Very satisfactory results were obtained.

Example VI

A concentrated solution of tetrachlophenol in propane was used for the impregnation of wood. Very satisfactory results were obtained.

Example VII

A 1% solution of phenyl mercury oleate in butane was found satisfactory for the impregnation of wood.

Example VIII

A 2% solution of alpha-nitronaphthalene in methyl chloride was satisfactorily employed, according to the above procedures, for the treatment of wood.

It will readily be understood that numerous

modifications may be made in the above illustrative examples without departing from the spirit of my invention. The impregnating agent used and its concentration will depend, of course, upon the characteristics which it is desired to impart to the wood. It is to be noted that other impregnating agents, such as alpha-nitronaphthalene and 2-4-dichlor-alpha-naphthol, may be used in propane or butane, and other agents, such as beta-naphthol and dinitrophenol may be used in methyl chloride.

The pressures of treatment may also be widely varied, and they will be determined for each case by the penetration desired, the nature of the wood, the temperature of treatment, and the length of treatment. When materials which can easily be impregnated are treated, it is, of course, unnecessary to use such high pressures, and the processes of my invention may, under some circumstances, advantageously be practiced at atmospheric pressures or at pressures only slightly above atmospheric.

It will be understood that the scheme of operation illustrated in the accompanying drawing may be widely varied without departing from the spirit of my invention. Instead of condensing the vaporized vehicle by means of refrigeration, the gas may be condensed by the use of a compressor. It will be readily apparent, moreover, that the processes already known for the impregnation of porous materials by the use of aqueous menstrooms and by the use of high boiling non-aqueous menstrooms may readily be adapted, according to the teachings of my invention, to the use of non-aqueous menstrooms which boil at temperatures no higher than 5° C.

While I have shown certain specific impregnating agents, certain non-aqueous menstrooms, and certain procedures and conditions of operation, it will be understood that I do not intend to be restricted thereby, the scope of my invention being apparent from the following claims.

I claim:

1. In a process for the impregnation of a porous material, the step comprising immersing the material in a liquid, non-aqueous menstroom which carries an impregnating agent, the non-aqueous liquid menstroom having a boiling point not substantially higher than about 1° C.

2. In a process for the impregnation of a porous material, the step comprising immersing the material in a liquid, non-aqueous menstroom in which is dissolved an impregnating agent, the non-aqueous liquid menstroom having a boiling point not substantially higher than about 1° C.

3. In a process for the impregnation of a porous material, the steps comprising immersing the material in a liquid, non-aqueous menstroom in which is dissolved an impregnating agent, the non-aqueous liquid menstroom having a boiling point not substantially higher than about 1° C., and after impregnation of the material, recovering the portion of the menstroom retained therein by volatilizing the menstroom therefrom.

4. In a process for the impregnation of wood, the step comprising immersing the material in a liquid, non-aqueous menstroom which carries an impregnating agent, the non-aqueous liquid hav-

ing a boiling point not substantially higher than about 1° C.

5. In a process for the impregnation of wood, the step comprising immersing the material in a liquid, non-aqueous menstroom in which is dissolved an impregnating agent, the non-aqueous liquid menstroom having a boiling point not substantially higher than about 1° C.

6. In a process for the impregnation of wood, the steps comprising immersing the material in a liquid, non-aqueous menstroom in which is dissolved an impregnating agent, the non-aqueous liquid menstroom having a boiling point not substantially higher than about 1° C., and after impregnation of the material, recovering the portion of the menstroom retained therein by volatilizing the menstroom therefrom.

7. In a process for the impregnation of wood, the steps comprising treating the wood by injecting thereinto under pressure an impregnating agent dissolved in a liquid, non-aqueous menstroom which has a boiling point not substantially higher than about 1° C. and, after impregnation of the wood, recovering the portion of the menstroom retained therein by volatilizing the menstroom therefrom.

8. In a process for the impregnation of wood, the steps comprising immersing the wood in a liquid, non-aqueous menstroom which has a boiling point not substantially above about 1° C., and in which is dissolved an impregnating agent, sealing the wood and liquid menstroom in a closed receptacle, raising the temperature of the menstroom to obtain a corresponding pressure within the receptacle, withdrawing the liquid menstroom from the receptacle, and volatilizing the portion of the menstroom retained in the wood to recover it therefrom.

9. In a process for the impregnation of wood, the steps comprising withdrawing air from the wood by preliminary vacuum, treating the wood under pressure with an impregnating agent dissolved in a liquid, non-aqueous menstroom which has a boiling point not substantially above about 1° C., and, after impregnation of the wood, recovering the portion of the menstroom retained therein by volatilizing the menstroom therefrom.

10. In a process for the impregnation of wood, the steps comprising impregnating the wood with a preservative dissolved in a liquid, non-aqueous menstroom which has a boiling point not substantially above about 1° C., and, after impregnation of the wood, recovering the portion of the menstroom retained therein by volatilizing the menstroom therefrom.

11. In a process for the impregnation of wood, the step comprising impregnating the wood by immersing it in liquid propane which carries an impregnating agent.

12. In a process for the impregnation of wood, the step comprising impregnating the wood by immersing it in liquid butane which carries an impregnating agent.

13. In a process for the impregnation of wood, the step comprising impregnating the wood by immersing it in liquid methyl chloride which carries an impregnating agent.

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