(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
12 April 2001 (12.04.2001)

PCT

(10) International Publication Number
WO 01/24982 A1

(51) International Patent Classification[7]: B27K 3/02, 3/08, 3/34

(21) International Application Number: PCT/SE00/01863

(22) International Filing Date:
26 September 2000 (26.09.2000)

(25) Filing Language: Swedish

(26) Publication Language: English

(30) Priority Data:
9903622-0 7 October 1999 (07.10.1999) SE

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(81) Designated States *(national)*: AE, AG, AL, AM, AT, AT (utility model), AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, CZ (utility model), DE, DE (utility model), DK, DK (utility model), DM, DZ, EE, EE (utility model), ES, FI, FI (utility model), GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KR (utility model), KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (utility model), SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:
— *With international search report.*
— *Before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments.*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 01/24982 A1

(54) Title: METHOD OF IMPREGNATION

(57) Abstract: A process for the impregnation of cellulose-based products, particularly wood, comprising treatment of the product with linseed oil at an increased temperature under pressure in an autoclave, comprising the steps: a) charging the autoclave with the product to be treated, b) charging the autoclave with linseed oil heated to a temperature exceeding the boiling point of water so that the product wil be surrounded by linseed oil, c) applying vacuum to the autoclave while keeping the temperature constant, water in the form of steam and air enclosed in the product being released from the product, d) discharging the linseed oil from the autoclave with simultaneous supply of linseed oil of a temperature lower than the boiling point of water and preferably applying over-pressure to the autoclave, and e) discharging the cold oil from the autoclave which, optionally, for the removal of excess oil from the product is again set under vacuum, whereafter the impregnated product is removed from the autoclave.

METHOD OF IMPREGNATION

The present invention relates to a process for linseed oil impregnation of a product based on cellulose, particularly wood, such as lumber, other wood products and the like. The process involves treatment of the product with linseed oil at an increased temperature under pressure in an autoclave.

Even if the present invention is applicable in relation to other cellulose-based products than wood the invention will in the following be described in connection with wood in the form of different types of lumber. Decomposition of wood when used outdoors is almost exclusively caused by fungi and bacteria. The requirement for growth of such micro-organisms is the presence of water. Furthermore, a suitable temperature and access to nutrition and oxygen are required. Prevention of decomposition can be based on the elimination of one or more of these conditions necessary for the growth of the micro-organisms. As an example there can be mentioned that if the moisture content is brought down to a value lower than about 20% biological decomposition is substantially prevented.

A number of different impregnation processes for the protection of wood are known, and these methods can in principle be divided up into techniques based on the supply of a substance poisonous to the growth of the micro-organisms, and techniques whereby the wood is given hydrophobic properties, for example by impregnation with some kind of oil. In the latter case an impregnation agent is crude linseed oil which can be of hot- or cold-pressed type, both of which by suitable heat treatment are converted into boiled linseed oils.

Most of the known methods for oil impregnation of lumber is based on the principle of using overpressure during the impregnation procedure. Moreover, impregnation

methods are known which are based on the so called principle "hot and cold-process", i.e. the lumber is initially treated in hot oil and is then transferred into cold oil whereby a certain improved impregnation result is obtained. The known methods are, however, unsatisfactory with regard to obtaining a degree of impregnation resulting in extended protection against attack by micro-organisms present in nature, for example micro-organism flora of aggressive brown rot.

The present invention has for an object to provide a process for linseed oil impregnation of cellulose-based products, particularly wood, the new process resulting in a substantially improved protective effect, especially in view of the fact that the process enables a higher level of uptake.

Another object of the invention is to provide a multi-step process utilizing an initial heating step in which water in the form of vapour and enclosed air is released from the product, and a subsequent step where the result of the water and air release is used to improve the uptake of oil.

Yet an object of the invention is to provide a process with alternate use of vacuum in combination with high temperature and then cooling in combination with over-pressure.

A particular object of the invention is to make certain in the process that the transition from the vacuum-heating step to the pressure-cooling step takes place continuously so that maximum benefit of this transition will be obtained.

For these and other objects which will be clear from the following description the invention provides for a process for the impregnation of cellulose-based products, particularly wood, with linseed oil, the products being treated with linseed oil at an increased temperature and under pressure in an autoclave. The invention is characterized by the following steps:

a) the autoclave is charged with the product to be treated,

b) linseed oil heated to a temperature exceeding the boiling point of water is introduced into the autoclave so that the product is surrounded by linseed oil,

c) while keeping the temperature at a constant level the autoclave is put under vacuum, water in the form of steam and air enclosed in the product being released from the product,

d) the linseed oil is discharged from the autoclave with simultaneous supply of linseed oil having a temperature lower than the boiling point of water and the autoclave is preferably put under overpressure, and

e) the cold oil is discharged from the autoclave which, optionally, for removal of excess oil from the product is again put under vacuum, the impregnated product then being removed from the autoclave.

The process according to the present invention is in principle based on the new concept that the switching over of the autoclave from hot linseed oil under vacuum takes place continuously by introducing cold linseed oil simultaneously with decharging the hot linseed oil from the autoclave, the autoclave being progressively put under overpressure. By this procedure the vacuum in the product generated under heat and autoclaved vacuum is utilized at a maximum so that optimal uptake of linseed oil takes place in connection with the supply of cold linseed oil and putting the autoclave under overpressure.

In the present disclosure the expressions "hot linseed oil" and "cold linseed oil" thus mean that the temperature of the oil in the first case exceeds the boiling point of water and in the latter case is lower than the boiling point of water, respectively.

In step a) it is suitable to heat the linseed oil to a temperature of about 140-180°C, whereas in the latter step c) it is preferred to put the autoclave under over-

pressure of up to about 15 bar, for example from about 5 to about 12 bar.

In the cooling step d) the temperature of the cold linseed oil is suitably maintained within the range about +75 to about +85°C.

In the process according to the present invention it is particularly preferred to use for the impregnation a processed linseed oil substantially consisting of linolenic acid, linolic acid and oleic acid, mainly in the form of triglycerides, the content of free tocoferol of the linseed oil being less than about 100 ppm. The linseed oil content of free tocoferol is suitably less than about 75 ppm and particularly less than about 50 ppm. Details regarding such processed linseed oil and the process for the manufacture thereof is found in patent application No. SE 9903621-2 filed simultaneously herewith with the same applicant and the same inventor. The contents of this co-pending patent application is incorporated herein by reference.

Finally, the process may in a further final step be dried in an air flow of environmental temperature.

The present invention also covers products, particularly wood products, obtained by the process described above.

The invention will in the following be further described in connection with no-limiting examples, wherein the percentages given relate to weight if not otherwise stated.

EXAMPLE 1

Impregnation procedure

In connection with the impregnation procedure there is used Swedish cold-pressed linseed oil from the harvest of 1998.

An autoclave is charged with lumber to be treated, in the present case rods which have been distributed and anchored. The autoclave is charged with linseed oil

heated to a temperature within the range about 140 to about 180°C, for example about 160°C, and the autoclave is charged so that the lumber is completely surrounded by linseed oil. The temperature is maintained at a constant level while the autoclave is put under vacuum. In view of the high temperature to which the lumber is heated and under the influence of vacuum the water will evaporize and water steam together with air will leave the pore system of the lumber.

After finished heat treatment which takes place for a period of about 1 h, the hot oil is continuously replaced with cold oil by pumping the hot oil out of the autoclave at the same time pumping cold oil into the autoclave at the bottom thereof. At the same time as this replacement of hot oil with cold oil the pressure in the autoclave is progressively increased from vacuum to an overpressure of about 10 bar. The temperature of the cold oil is maintained within the range about +75 to about +85°C. In cooling of the lumber a sub-pressure is created in the pore system thereof, whereby impregnating oil more easily impregnates the lumber so as to increase the level of uptake.

After finished treatment in the autoclave the lumber is allowed to dry in a cool flow of air at a temperature of about 20°C or lower, whereby excess of oil further penetrates into the lumber. This final step has for a function to prevent that oil by later sweating emerges from the impregnated lumber.

EXAMPLE 2

Result of impregnation

The lumber made subject to impregnation can be of any type. In Sweden the trend is presently towards the common coniferous trees, namely fir and pine. The lumber can be fresh or dried in different degrees, i.e. contain different amounts of moisture.

The time periods of the different process steps will have to be adapted to the parameters of the lumber (dimension, moisture content, type of wood, core and/or sapwood) and to the uptake level desired. The uptake level can be given in quantity of linseed oil per cubic meter or percent of the dry density of the lumber which is about 450 kg/m^3, and further with regard to the intended use of the impregnated lumber. The higher the uptake level obtained the better resistance of the lumber and the harder conditions endured by lumber in its practical use.

Different types of lumber have been test impregnated in accordance with the process of the invention, and in the following table these types of lumber and the uptake levels obtained are given.

TABLE

Type of lumber (density 450 kg/m^3)	Approximate uptake level %	About kg/m^3
Pine sap	100%	450 kg/m^3
Pine core	40%	180 kg/m^3
Fir sap	40%	180 kg/m^3
Fir core	20%	90 kg/m^3

Lumber impregnated to high levels of uptake, i.e. up to 100%, can according to ongoing tests endure long periods of time under quite severe conditions, i.e. freely outdoors in ground or in ground contact.

It should be observed that the present invention is not restricted to the specific embodiments exemplified above. Thus, modifications and changes can be carried out within the frame-work of the invention and such changes and modifications are easily understood by those skilled in the art.

CLAIMS

1. A process for the impregnation of cellulose-based products, particularly wood, comprising treatment of the product with linseed oil at an increased temperature under pressure in an autoclave, characterized by the steps:

a) charging the autoclave with the product to be treated,

b) charging the autoclave with linseed oil heated to a temperature exceeding the boiling point of water so that the product will be surrounded by linseed oil,

c) applying vacuum to the autoclave while keeping the temperature constant, water in the form of steam and air enclosed in the product being released from the product,

d) discharging the linseed oil from the autoclave with simultaneous supply of linseed oil of a temperature lower than the boiling point of water and preferably applying over-pressure to the autoclave, and

e) discharging the cold oil from the autoclave which, optionally, for the removal of excess oil from the product is again set under vacuum, whereafter the impregnated product is removed from the autoclave.

2. A process according to claim 1, characterized in that in step a) the linseed oil is heated to a temperature of about 140 to 180°C.

3. A process according to claim 1 or 2, characterized in that in step c) the autoclave is set under an over-pressure of up to about 15 bar.

4. A process according to any one of the preceding claims, characterized in that in step d) the temperature of the cold linseed oil is maintained within the range about 75 to 85°C.

5. A process according to any one of the preceding claims, characterized in that in step d) the over-pressure is from about 5 bar to about 12 bar.

6. A process according to any one of the preceding claims, characterized in that the impregnation is carried out with a processed linseed oil substantially consisting

of linolenic acid, linolic acid and oleic acid, mainly in the form of triglycerides, the contents of the oil of free tocoferol being less than about 100 ppm.

7. A process according to claim 6, characterized in that the contents of free tocoferol of the linseed oil is less than about 75 ppm.

8. A process according to claim 7, characterized in that the contents of free tocoferol of the linseed oil is less than about 50 ppm.

9. A process according to any one of the preceding claims, characterized in that the product in a further final step is dried in an air flow of environmental temperature.

10. Products produced by the process according to any one of the preceding claims.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 00/01863

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: B27K 3/02, B27K 3/08, B27K 3/34
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: B27K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3968276 A (WILLIAM R. ALLEN), 6 July 1976 (06.07.76)	1-10
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A	US 5652023 A (ANTHONY J. BERGERVOET ET AL), 29 July 1997 (29.07.97)	1-10
	--	
A	WO 9846403 A1 (BWG BUTZBACHER WEICHENBAU GMBH), 22 October 1998 (22.10.98)	1-10
	--	
A	WO 9411167 A1 (HUNGBAU KFT.), 26 May 1994 (26.05.94)	1-10
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[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
2 February 2001	05 -02- 2001
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Solveig Gustavsson/EÖ Telephone No. +46 8 782 25 00

Form PCT/ISA/210 (second sheet) (July 1998)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 00/01863

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 9219429 A1 (SCHIRNIG, ULRICH, HERBERT), 12 November 1992 (12.11.92) -- --------	1-10

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/SE 00/01863

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US 3968276 A	06/07/76	NONE	
US 5652023 A	29/07/97	AU 1979497 A	16/09/97
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		EP 0576608 A,B	05/01/94