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(54) **METHOD OF IMPREGNATION**

IMPREGNIERUNGSVERFAHREN

PROCEDE D'IMPREGNATION

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(56) References cited:
WO-A1-92/19429 **WO-A1-94/11167**
WO-A1-98/46403 **US-A- 3 968 276**
US-A- 5 652 023

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Description

[0001] 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.

[0002] 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.

[0003] 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.

[0004] 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.

[0005] 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.

[0006] 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.

[0007] 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.

[0008] 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.

[0009] 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.

[0010] 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 discharging 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.

[0011] 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.

[0012] In step b) it is suitable to heat the linseed oil to a temperature of about 140-180°C, whereas in the latter step

d) 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.

[0013] In the cooling step d) the temperature of the cold linseed oil is suitably maintained within the range about +75 to about +85°C.

[0014] 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.

[0015] Finally, the process may in a further final step be dried in an air flow of environmental temperature.

[0016] 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

[0017] In connection with the impregnation procedure there is used Swedish cold-pressed linseed oil from the harvest of 1998.

[0018] 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.

[0019] At the end of the 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.

[0020] At the end of the 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

[0021] 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.

[0022] 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 sap-wood) 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/m³, 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.

[0023] 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

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TABLE (continued)

Type of lumber (density 450 kg/m ³)	Approximate uptake level %	About kg/m ³
Fir core	20%	90 kg/m ³

5 [0024] 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.

10 [0025] 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

15 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, comprising the steps of:

- 20 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,
25 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.

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

35 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.

40 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.

45 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.

50 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.

Patentansprüche

55 1. Verfahren zum Imprägnieren von Produkten auf Cellulosebasis, insbesondere von Holz, bei dem das Produkt mit Leinöl bei erhöhter Temperatur unter Druck in einem Autoklaven behandelt wird, wobei das Verfahren die Stufen

umfasst:

- 5 a) Einführen des zu behandelnden Produkts in einen Autoklaven,
b) Einführen von Leinöl, das auf eine Temperatur erhitzt ist, die den Siedepunkt von Wasser übersteigt, in den Autoklaven, sodass das Produkt von Leinöl umgeben ist,
c) Anlegen eines Vakuums an den Autoklaven, während die Temperatur konstant gehalten wird, wobei Wasser in Form von Wasserdampf und Luft, die in dem Produkt eingeschlossen ist, aus dem Produkt freigesetzt werden,
10 d) Austragen des Leinöls aus dem Autoklaven unter gleichzeitiger Zufuhr von Leinöl mit einer Temperatur unterhalb des Siedepunkts von Wasser und vorzugsweise unter Anlegen eines Überdrucks an den Autoklaven und
e) Austragen des kalten Öls aus dem Autoklaven, der gegebenenfalls zur Entfernung von überschüssigem Öl aus dem Produkt erneut unter Vakuum gesetzt wird, wonach das imprägnierte Produkt aus dem Autoklaven entnommen wird.
- 15 2. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, dass** in der Stufe (b) das Leinöl auf eine Temperatur von etwa 140 bis 180 °C erhitzt wird.
- 20 3. Verfahren nach Anspruch 1 oder 2, **dadurch gekennzeichnet, dass** in der Stufe (d) der Autoklav unter einen Überdruck von bis zu etwa 15 bar gesetzt wird.
4. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** in der Stufe (d) die Temperatur des kalten Leinöls innerhalb des Bereiches von etwa 75 bis 85 °C gehalten wird.
- 25 5. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** in der Stufe (d) der Überdruck etwa 5 bis etwa 12 bar beträgt.
- 30 6. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** die Imprägnierung mit einem behandelten Leinöl durchgeführt wird, das im Wesentlichen aus Linolensäure, Linolsäure und Ölsäure, hauptsächlich in Form der Triglyceride, besteht, wobei der Gehalt des Öls an freiem Tocopherol weniger als etwa 100 ppm beträgt.
- 35 7. Verfahren nach Anspruch 6, **dadurch gekennzeichnet, dass** der Gehalt des Leinöls an freiem Tocopherol weniger als etwa 75 ppm beträgt.
8. Verfahren nach Anspruch 7, **dadurch gekennzeichnet, dass** der Gehalt des Leinöls an freiem Tocopherol weniger als etwa 50 ppm beträgt.
- 40 9. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** das Produkt in einer weiteren Schlussstufe in einem Luftstrom von Umgebungstemperatur getrocknet wird.

Revendications

- 45 1. Procédé d'imprégnation de produits à base de cellulose, particulièrement du bois, comprenant le traitement du produit avec de l'huile de lin à une température élevée sous pression dans un autoclave, comprenant les étapes suivante :
- 50 a) chargement de l'autoclave avec le produit à traiter,
b) chargement de l'autoclave avec de l'huile de lin chauffée à une température dépassant le point d'ébullition de l'eau de sorte que le produit soit entouré d'huile de lin,
c) application d'un vide à l'autoclave tout en maintenant la température constante, de l'eau sous la forme de vapeur d'eau et de l'air renfermé dans le produit étant libéré du produit,
55 d) déchargement de l'huile de lin de l'autoclave avec une alimentation simultanée en huile de lin d'une température inférieure au point d'ébullition de l'eau et, de préférence, application d'une surpression à l'autoclave, et
e) déchargement de l'huile froide de l'autoclave qui, éventuellement, pour l'élimination de l'huile excédentaire du produit, est de nouveau soumis à un vide; après quoi, le produit imprégné est retiré de l'autoclave.

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2. Procédé selon la revendication 1, **caractérisé en ce que**, à l'étape b), l'huile de lin est chauffée à une température d'environ 140°C à 180°C.
- 5 3. Procédé selon la revendication 1 ou 2, **caractérisé en ce que**, à l'étape d), l'autoclave est soumis à une surpression jusqu'à environ 15 bars.
4. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que**, à l'étape d), la température de l'huile de lin froide est maintenue dans la plage d'environ 75°C à 85°C.
- 10 5. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que**, à l'étape d), la surpression se situe dans la plage d'environ 5 à environ 12 bars.
- 15 6. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que** l'imprégnation est effectuée avec une huile de lin traitée constituée sensiblement de l'acide linoléique, de l'acide linoléique et de l'acide oléique, principalement sous la forme de triglycérides, la teneur en huile de tocophérol libre étant inférieure à environ 100 ppm.
- 20 7. Procédé selon la revendication 6, **caractérisé en ce que** la teneur en tocophérol libre de l'huile de lin est inférieure à environ 75 ppm.
- 25 8. Procédé selon la revendication 7, **caractérisé en ce que** la teneur en tocophérol libre de l'huile de lin est inférieure à environ 50 ppm.
- 30 9. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que** le produit, dans une autre étape finale, est séché dans un flux d'air à température environnementale.

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