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(54) **METHOD FOR INCREASING THE RESISTANCE OF CELLULOSIC PRODUCTS AGAINST MOULD AND DECAY**

VERFAHREN ZUR VERBESSERUNG DES WIDERSTANDES VON ZELLULOSEHALTIGEN PRODUKTEN GEGEN SCHIMMEL UND FÄULNIS

PRODEDE PERMETTANT D'AMELIORER LA RESISTANCE A LA MOISSURE ET POURRITURE DE PRODUITS CELLULOSIQUES

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Description

[0001] The present invention concerns a method according to the preamble of claim 1 for improving the resistance of cellulosic products against mould and decay as well as to enhance the dimensional stability of the products.

[0002] According to a method of the present kind the cellulosic product is dried to a moisture content of less than 15% and then subjected to a heat treatment carried out at an elevated temperature.

[0003] It is well-known in the art that the dimensional stability of wood can be improved by heat treatments. As far as the prior art is concerned, reference is made to, for instance, the Finnish Patent Specification No. 68,122, which discloses a method for treating wood products at temperatures of 160 to 240 °C and at pressures of 3 to 15 bar. As a result of the treatment, the capability of wood to absorb water and thus to expand is considerably reduced. The effect of heat treatments on decay resistance of wood has also been studied. Mailun, N.P. and Arenas, C.V describe in their article "Effect of heat on natural decay resistance of Philippinean woods" (Philippinen Lumberman, Vol 20, No. 10, 1974, p. 18-19, 22-24) the treatment of Asian wood species in dry state at temperatures of 90, 110, 130, 150 and 175 °C for 240 hours. As a result of the treatment the colour of the wood changes to chocolate brown. An extended treatment at 130, 150 and 175 °C increased the resistance of the wood samples against two brown rot fungi. However, at the same time it made the wood weaker.

[0004] Because all kinds of wood are not suited for the conventional pressure impregnation methods, using substances which prevent the growth and spreading of fungi, heat treatment is an interesting alternative for protecting wood against decay.

[0005] The prior heat treatment processes, which call for the use of pressure and extended treatment times, have been too complicated for industrial applicability. It has also been ascertained that under the influence of high pressures and increased temperatures wood becomes brittle and it weakens. Furthermore, wood is easily ignited at high temperatures.

[0006] The present invention aims at eliminating the problems related to the prior art and to provide a completely novel solution for improving the dimensional stability of and the resistance against decay and mould (i.e. the biodegradation resistance) of cellulosic products.

The invention is based on the concept of carrying out the heat treatment of the cellulosic product in two stages: first the product is dried to desired moisture content, typically to below 15%. Then the temperature is rapidly raised above 150°C (typically to about 180 to 250°C) and the treatment is continued at that temperature until the weight loss of the treated product amounts to at least 5%. The products are kept at essentially atmospheric pressure in a moist oven, to which water steam is fed. By contrast, DE-A-2 263 758 mentioned above, suggests working at pressures of 5 and 6.5 and the weight loss achieved by the known method is small. Similarly, it is an objective of WO-A-90/06840 to avoid weight loss of wood caused by heating at elevated temperatures.

In particular the method according to the invention is principally characterized by what is stated in the characterizing part of claim 1.

[0007] As mentioned above, unseasoned timber or similar cellulosic products are used as starting materials for the method according to the present invention. The product can be dried at any suitable conditions (even outdoors at ambient temperature) to the desired moisture content of less than 15 %. According to a preferred embodiment of the invention the product is, however, dried at elevated temperatures. The colour of the wood product will become darker during such drying. In connection with the drying due care is taken to avoid cracking of the product. This goal is advantageously attained by constantly determining the temperatures of the interior and the surface, respectively, of the wood and by maintaining the temperature difference at a reasonably small value. Preferably said difference amounts to about 10 to 30 °C. This procedure is followed both when the temperature is raised and when it is lowered. Surprisingly, it has been found that said solution will even completely prevent the formation of cracks in (the interior parts of) the wood material. When larger amounts of wood are to be dried several samples should be provided with sensors. On an industrial scale, the preferred procedure comprises determining for each kind of timber a heating programme of its own which takes into account the influence of the initial moisture content on the process.

[0008] In order to protect the wood and improve heat transfer during drying it is preferred to use steam during drying. According to a preferred embodiment, the drying process according to the invention comprises the following steps:

a) first the temperature of the drying oven is raised to at least about 90 °C, preferably to at least 100 °C, and that temperature is maintained until the wood has at least approximately reached the same temperature,
b) then the temperature of the oven is gradually raised so that the difference between the interior temperature of the wood and the temperature of the oven does not exceed 30 °C until the desired moisture content of the wood is reached, and
c) finally, the temperature of the oven is lowered gradually while ensuring that the difference between the temperature of the interior parts of the wood and the temperature of the oven does not exceed 30 °C until the interior of the wood has reached the desired temperature.

[0009] If needed, stage c can be left out. The heat treatment, which will be described in more detail below, is then carried out immediately after stage b.

[0010] During the first stage of the present invention (step a), the temperature of the drying oven is preferable set at a value of about 100 to 150 °C, preferably 100 to 120 °C. In the second stage (step b), the heating is stopped when the humidity of the wood is below 15 %, e.g. 1 to 15 %. During step b and step c, if any, the difference between the external temperature and the interior temperature of the cellulosic product is kept at a value of 10 to 30 °C. Too small a temperature difference prolongs the drying process, whereas too large a difference increases the risk of internal cracking. During stage c the temperature of the oven is lowered until the interior temperature of the wood has decreased below 100 °C.

[0011] During stages a, b, and c water steam is fed into the oven to keep the wet temperature at about 80 to 120 °, preferably at about 100 °C. It is preferred to use saturated water steam.

[0012] When the moisture content of the product has dropped to below 15 %, as a result of the drying, the treatment is continued at an elevated temperature.

[0013] During the second stage of the process the temperature is kept higher than during the first stage of the process. It is preferred to operate the process at about 180 to 250 °C in an atmosphere of saturated steam. The temperature can also be raised during the second stage, as will appear from Example 2. The duration and the temperature of the treatment are interdependent, as explained in connection with Example 1. Typically, the heat treatment of the second stage takes at least some 0.5 hours, preferably 1 to 20 hours and in particular about 2 to 10 hours. The weight loss of the product can be adjusted by varying the heat treatment. This makes it possible to change the strength and decay resistance properties of the product as desired. Therefore, the heat treatment is continued until a weight loss of at least 5% (based on dry matter) has been obtained. Clear improvements of the dimensional stability of the product are reached at this value already. Mould and decay resistance will also be improved, and further improvements of said properties can be obtained by continuing the heating until at least about 6 or even 8%, weight loss has taken place in the product.

[0014] Summarizing, the features obtained by the present invention are:

- Improvement of decay resistance (in comparison to wood which natively has a good resistance to decay)
- Improvement of mould resistance
- Improvement of the dimensional stability
- Removal of pitch
- Heat conductivity decreased by 25 - 40 %
- Improvement of paint adherence

[0015] The heat treatment of the second stage is, according to a preferred embodiment of the invention, carried out at least essentially under non-pressurized conditions, i.e. at atmospheric pressure.

[0016] The method according to the invention is suited for treatment massive wood goods, such as logs and pillars. In addition, the method can be applied to veneer, chips, saw dust, wood fibres and other cellulosic products, such as, for instance, crates.

The wood preservation effect that can be produced is studied in more detail in Example 2. However, in this connection it should be pointed out that good protection against decay requires that dried sawn timber of pine is kept for preferably about 2 to 8 hours at a temperature of 200 to 250 °C. The same conditions are used for birch and larch-tree, whereas good protection against decay can be obtained at slightly lower temperatures for spruce. Thus, spruce can be treated, for instance, at about 175 to 210 °C. The method is well-suited for treatment of aspen.

[0017] Example 3 explains in detail the decrease of heat conductivity as a result of a treatment carried out according to the invention.

[0018] The invention provides considerable advantages. It will therefore provide for a shortening of the time required for drying of wood. The colour changes appearing during drying can be utilized and, at the same time, the resistance of wood against decay and mould and the dimensional stability can be improved. Detrimental pitch can be removed from samples of coniferous wood by the treatment. As examples of products that can be treated with the method according to the present invention, the following can be mentioned: external cladding, window frames, outdoor furniture, and boards for sauna platforms.

[0019] After a treatment according to the present invention, the dimensional instability under the influence of moisture is reduced by 50 to 70 %. The resistance against decay of the products is improved. At its best, the resistance is on the same level as that obtained by pressure impregnation or even better without any substantial weakening of the strength properties of the products. The treated wood forms a good surface for paint.

[0020] The preparation process is simple and quick (short treatment times) and there is no need to use pressure. As far as its weathering resistance, resistance to decay and mould, and strength properties are concerned, the product can be modified in a controlled manner by the method. The method is suitable for all kinds of wood. By means of the heat treatment it is become possible also to improve the properties of the heartwood, which cannot be done by pressure impregnation. The durability of those kinds of wood which are difficult to impregnate can be improved. The improvement of the permeability of wood makes it possible to impregnate the wood with other colouring agents.

[0021] In the following the invention will be examined in greater detail with the help of the attached drawings and some working examples.

Figure 1 is a simplified schematic representation of the construction of an apparatus which can be used for carrying out the present invention,
Figure 2 indicates the influence of the temperature and treatment time on the weight loss of the product,
Figure 3 indicates the reduction of tangential swelling of the wood sample as a function of the weight loss,
Figure 4 indicates the reduction of radial swelling of the wood sample as a function of the weight loss,
Figure 5 indicates the reduction of moisture taken up by the wood sample as a function of the weight loss,
Figure 6 indicates the changes of bending strength caused by the heat treatment,
Figure 7 shows the moisture contents of bending test samples after conditioning for 4 weeks,
Figure 8 shows the weight losses of heat treated and control samples, respectively, after decay testing,
Figure 9 shows the drying of unseasoned spruce according to a preferred embodiment according to the present invention,
Figure 10 indicates the weight losses of veneer as a function of the duration of the heat treatment,
Figure 11 indicates the reduction of thickness swelling of plywood as a result of a heat treatment, and
Figure 12 indicates the reduction of the moisture content of plywood under the influence of a heat treatment.

[0022] An apparatus shown, for instance, in Figure 1 is used in the present invention. The apparatus comprises an oven 2 surrounded by an oven jacket 1. The samples 3 are placed in the oven, which is provided with inlet 4 and outlet 5 channels for air 5, for conducting moist air through the oven. The outlet channel 5 is combined with a steam feed pipe 6 for feeding more water steam into the outlet air coming from the oven. In order to form a closed cycle the inlet and outlet channels are joined each to its end of a set of ducts 7 provided with a fan 9 and with heating means 8. The air flowing through said ducts are heated by electric resistances 8 to the set temperature and conducted via the fan 9 to the inlet channel 4 of the oven. The recycling direction of the air in the apparatus is indicated with an arrow.

[0023] By using the present apparatus it is possible to make sure that the samples placed in the oven are heated to the desired temperature by moist air. By changing the amount of steam, which is being fed, the moisture content of the air can be altered. Usually, the air of the oven is saturated with water steam.

Example 1

Heat treatment of wood

[0024] Moist wood is dried in the above-described apparatus at 120 to 140 °C either with steam or without it. As a result of the treatment, there is some darkening of the colour of the wood sample, but no cracking. When the moisture content of the wood is below 15 % the temperature is raised to at least 175 °C, preferably to 180 to 250 °C. The treatment is continued for 2 to 10 hours. Saturated steam is conducted to the apparatus. By varying the temperature and the time, the desired result can be obtained. The colour of the wood darkens further.

[0025] Figure 2 shows the influence of temperature and time on the reduction of wood weight.

[0026] By adjusting the weight losses the properties of the wood can be changed as desired. Figures 3, 4 and 5 depict the reduction of tangential swelling of the wood, the reduction of the radial swelling of the wood, and the reduction of the amount water absorbed by the wood (wood moisture content) in comparison to the control samples. The graphs of Figures 4 and 5 correspond to graph model of Figure 1.

[0027] The heat treatment weakens the bending strength of wood after a certain weight loss. On the other hand, the experiments show that the bending strength properties of some of our samples were even better than the corresponding properties of the control samples (Figure 6). This is due to the fact that, depending on ambient humidity, some of the heat treated samples clearly adsorbed less water than the control sample (Figure 7).

Example 2

Decay test

[0028] The decay test was carried out according to European Standard EN 113 modified as follows: the number of parallel test specimens was four, the sizes of the test specimens were 5x20x35 mm, and they were not rinsed before the test. The samples were subjected to the test rot fungus, cellar fungus (*Coniophora puteana*), for 2, 4, 8, and 12 weeks.

[0029] The test specimens were sawn from planks of pine, birch, larch-tree and spruce, treated according to example 1. Table 1 contains a summary of the conditions prevailing during the heat treatment.

Table 1.

Treatment conditions of the test specimens of the decay test		
Sample	Wood species	Heat treatment (time/temp.)
1	Pine	1.5 h/200 °C + 1 h /220 °C
2	Pine	2 h/220 °C
3	Pine	4 h/210 °C
4	Birch	1 h/160 °C
5	Birch	4 h/210 °C
6	Birch	1 h/160 °C + 2 h/220 °C
7	Birch	1 h/160 °C + 4 h/220 °C
8	Larch-tree	4 h/210 °C
9	Spruce	4 h/210 °C
10	Spruce	1 h/180 °C

[0030] After the heat treatment the dry matter of the wood specimens were determined. The test specimens were sterilized by radiation (Co-60), the sterilized test specimens were inserted in kolle dishes on a fungus culture growing on malt agar medium. At least one heat treated test specimen and one untreated control sample were inserted into each dish.

[0031] At the end of the decay test the specimens were dried at 103 °C and the weight losses of the specimens were calculated according to EN 113. For pine a weight loss of less than 10 % was achieved by the heat treatment; the weight losses for untreated wood were over 30 %. The smallest weight losses for heat treated birch, larch-tree and spruce were close to zero.

[0032] The results of the decay tests are indicated in Figure 8. It is apparent from the figure that a mild heat treatment (160 °C) does not yet significantly improve the decay resistance of the timber.

Example 3

Drying of unseasoned spruce

[0033] When a specimen of wet spruce (50 x 100 x 1500 mm), initial moisture content about 40 %, was heated according to the preferred drying embodiment of the invention for 24 hours by operating the drying system in such a way that the difference between the internal and external temperatures was 10 to 20 degrees, no cracks were found in the test specimen (Figure 9). The final moisture of the dry test specimen was below 5 %.

Example 4

Reduction of heat conductivity

[0034] Table 2 presents the heat conductivities of heat treated samples of spruce, pine and aspen. The table also indicates the conditions of the heat treatment.

Table 2.

Heat conductivity of the test specimens		
Wood/treatment, temperature and time	Density at time of measuring, kg/m^3	Heat conductivity λ_{10}, W/(mK)
Aspen, control	415	0.098
Aspen, 4 h 210 °C	403	0.077
Aspen, 10 h 210 °C	379	0.077

Table 2. (continued)

Heat conductivity of the test specimens		
Wood/treatment, temperature and time	Density at time of measuring, kg/m^3	Heat conductivity λ_{10}, W/(mK)
Spruce, control	497	0.11
Spruce, fresh, 26 h, heat treatment: 3 h / 220 °C	375	0.086
Spruce, 8 h 230 °C	399	0.080
Pine, control	583	0.13
Pine, fresh, heat treatment: 3 h 220 °C	520	0.107
Pine, 30 h 230 °C	476	0.088

Example 5

[0035] Birch veneer, thickness 1.5 mm, was heat treated in an oven of the kind shown in Figure 1. The temperature of the treatment was 200 °C and the time 2 to 7 hours.

[0036] The test specimens were selected by dividing the veneer into two parts and by choosing one half of the veneer as a control. The other half was heat treated. A 3-ply plywood was prepared from the veneer. The gluing was made by FF glue, which was applied to the surfaces of the veneer by a brush. The veneers were pressed together at 130 °C for 6 minutes. The compression load was 1.7 MPa. The control plywood and the plywood prepared from the heat treated veneers were kept in the same pressing.

[0037] In order to determine the thickness swelling, the test specimens were dried in an oven at 102 °C. Then they were immersed into 20 °C water for 2, 6, 26, and 168 hours. The test specimens were prepared for the strength testing by conditioning them at a relative humidity of 65 %, whereinafter they were evaluated for wood failure, tensile strength and bending strength. The tests included two parallel test specimens.

[0038] The weight loss of the wood (calculated on basis of the dry matter) caused by the heat treatment is indicated in Figure 10. As a result of the treatment the weight of the wood decreased by 3.4 to 8.4 %.

[0039] The thickness swelling of the plywood is indicated in Figure 3.

Table 3.

Thickness swelling of plywood and moisture content of plywood after immersion into water								
Sample	Thickness swelling				Moisture content			
	2 h	6 h	27 h	168 h	2 h	6 h	27 h	168 h
2 h heat	2.9	5.5	10.2	11.7	18.0	29.6	50.0	79.1
2 h control	9.1	11.4	12.3	12.5	38.0	49.5	60.8	72.3
3 h heat	2.7	5.2	8.9	11.0	14.8	27.0	43.2	74.8
3 h control	5.5	9.3	10.9	11.1	28.8	43.5	61.4	75.9
4 h heat	1.8	3.7	7.4	9.9	13.4	23.6	42.0	74.2
4 h control	8.6	11.9	14.5	14.7	28.8	39.3	53.9	66.5
5 h heat	1.9	4.1	8.0	11.2	16.1	27.4	46.6	79.9
5 h control	6.8	12.2	10.9	11.1	34.1	47.3	65.6	78.7
6 h heat	1.8	3.6	6.3	8.6	14.5	25.1	43.0	77.2
6 h control	5.4	8.6	10.4	10.6	29.1	43.3	62.8	79.0
7 h heat	1.2	2.8	5.5	7.9	13.2	22.2	38.0	67.5
7 h control	6.7	9.7	11.1	11.2	31.2	48.8	59.1	73.7

[0040] The thickness swelling of the control samples varied to a large extent. For this reason, the swelling reduction results presented in Figure 11 have been calculated in relation to the control samples of each test series. Figure 12 shows the reduction of the amounts of water absorbed by the wood samples compared to the untreated samples.

[0041] As far as thickness swelling is concerned the best results were obtained by the treatment having the longest duration, i.e., by a 7 hour heat treatment. After a 2 hour immersion the thickness swelling was then 80 % smaller than that of the control samples. An almost equally good a result was reached by a 4 hour treatment. 2 and 3 hour heat treatments reduced thickness swelling after a 2 hour immersion to 50 or 70 %. After a 24 hour immersion the thickness swelling of plywood which had been heat treated for 7 and 4 hours was 50 % smaller than that of the control samples.

[0042] The heat treatment reduces the amount of water absorbed by the wood sample (= moisture content of wood). Subject to immersion into water for 24 hours, the moisture content of plywood which had been heat treated for 7 hours was about 38 % smaller than that of the control plywood.

[0043] Table 4 indicates the strength properties of the plywood articles.

Table 4.

Strength properties of the plywood					
Sample	Shear strength of glue line		Tensile strength N/mm^2	Moisture content (%) during strength testing	Bending strength /mm^2
	Wood failure %	Resist, to shear N/mm^2			
2 h heat	21	1.7	65.3	5.0	132
2 h control	97	3.0	81.7	5.3	148
3 h heat	81	2.4	83.1	4.7	155
3 h control	98	3.0	101.7	4.1	148
4 h heat	99	2.2	55.4	5.2	130
4 h control	95	3.0	110.9	5.0	165
5 h heat	100	1.7	50.4	4.2	95
5 h control	84	3.1	74.5	4.7	128
6 h heat	99	1.9	37.5	4.5	108
6 h control	77	2.4	92.8	4.9	139
7 h heat	97	1.8	55.2	4.9	101
7 h control	94	2.9	84.5	5.4	141

[0044] Requirements for a 3-ply plywood:

- Shear strength of glue line, dry, strength = 2.1 N/mm^2. If the strength is less than that, the wood failure percentage should be more than or equal to 50 %.
- Tensile strength 54 N/mm^2
- Bending strength 72 N/mm^2

[0045] The tensile strength of plywood prepared from heat treated veneer was almost always less than the required 2.1, but because the wood failure % exceeded 50, it should be noted that the requirements regarding shearing strength were nevertheless fulfilled.

[0046] The bending strength of the plywood prepared from heat treated veneer was inferior to that of the control plywood, but even so it met the requirements. The required bending strength was not reached with heat treated veneer which had been heat treated for 5 or 6 hours.

Example 6

Field trials

[0047] Test specimens (50 x 25 x 500 mm) were heat treated for 4 hours at 220 °C. The samples were placed on test field in contact with the earth. After a time of one year the test specimens were checked and evaluated.

[0048] The results were evaluated using the following scale: 1 = some beginning decay (25 %), 2 = 50 %, 3 = 75 %, 4 = the test specimen breaks under a weight. Average values of the results:

Pine, control = 0.3. Heat treated pine = 0.
Spruce control = 1. Heat treated spruce = 0.2.
Birch, control = 3.6. Heat treated birch = 2.5.

Claims

1. A method for increasing the resistance of cellulosic products against mould and decay and for improving the dimensional stability of the product, according to which method

- the cellulosic products are dried to a moisture content level of less than 15 %,
- they are subjected to a heat treatment carried out at an increased temperature,

characterized in that

- after drying the products are kept at essentially atmospheric pressure at a temperature above 150 °C in a moist oven, to which water steam is fed, and
- the treatment is continued until a weight loss of at least 5 % has taken place in the product.

2. A method according to claim 1, wherein the cellulosic products are dried at an elevated temperature, **characterized** in that during drying of the wet product the difference between the inner and the outer temperatures of the product is maintained at about 10 to 30 °C in order to prevent cracking.

3. A method according to claim 2, **characterized** in that the product is dried in the presence of steam.

4. A method according to claim 2 or 3, **characterized** in that the drying of the product comprises the steps of

a) placing it in a drying oven, whose temperature is raised to at least 90 °C, preferably to at least 100 °C, and the oven is kept at this temperature until the cellulosic product has at least approximately reached the same temperature,
b) subsequently gradually increasing the temperature of the oven while maintaining the difference between the inner temperature of the products and the temperature of the oven at less than 30 °C until the desired moisture content of the wood has been reached and finally
c) optionally gradually lowering the oven temperature while keeping the difference between the inner temperature of the wood and the temperature of the oven at less than 30 °C until the inner temperature of the wood has reached the desired value.

5. A method according to any of the preceding claims, **characterized** in that a cellulosic product dried to a moisture content of less than 15 % is treated at 180 to 250 °C for 1 to 20 hours, preferably for about 2 to 10 hours.

6. A method according to any of the preceding claims, **characterized** in that saturated water steam is fed to the process.

7. A method according to claim 1, **characterized** by treating wood, pillars, logs, sawn wood, veneer, plywood, chips, saw dust, or fibres.

8. A method according to claim 1, wherein sawn pine wood is treated, **characterized** by keeping the dried sawn wood at a temperature of 200 to 250 °C for 2 to 8 hours so as to achieve good protection against decay.

9. A method according to claim 1, wherein sawn spruce wood is treated, **characterized** by keeping the dried sawn wood at a temperature of 175 to 210 °C for 2 to 8 hours so as to achieve good protection against decay.

10. A method according to claim 1, wherein birch wood material is treated, **characterized** by keeping the dried sawn wood at a temperature of 200 to 250 °C for 2 to 8 hours so as to achieve good protection against decay.

11. A method according to claim 1, wherein larch-tree wood material is treated, **characterized** by keeping the dried sawn wood at a temperature of 200 to 250 °C for 2 to 8 hours so as to achieve good protection against decay.

Patentansprüche

1. Verfahren zur Erhöhung der Beständigkeit von Celluloseprodukten gegenüber Schimmel und Fäulnis und zur Verbesserung der Formbeständigkeit des Produkts, wobei gemäß diesem Verfahren:

- die Celluloseprodukte auf einen Feuchtigkeitsgehalt von weniger als 15% getrocknet werden,
- sie einer Wärmebehandlung unterworfen werden, die bei erhöhter Temperatur durchgeführt wird,

dadurch gekennzeichnet, dass:

- die Produkte nach dem Trocknen im Wesentlichen bei Atmosphärendruck und einer Temperatur über 150°C in einem Feuchtofen, der mit Wasserdampf beschickt wird, gehalten werden, und
- die Behandlung so lange fortgesetzt wird, bis im Produkt ein Gewichtsverlust von mindestens 5% stattgefunden hat.

2. Verfahren nach Anspruch 1, wobei die Celluloseprodukte bei erhöhter Temperatur getrocknet werden, dadurch gekennzeichnet, dass beim Trocknen des feuchten Produkts der Unterschied zwischen Innen- und Außentemperatur des Produkts bei etwa 10 bis 30°C gehalten wird, so dass ein Reißen verhindert wird.

3. Verfahren nach Anspruch 2, dadurch gekennzeichnet, dass das Produkt in Anwesenheit von Dampf getrocknet wird.

4. Verfahren nach Anspruch 2 oder 3, dadurch gekennzeichnet, dass das Trocknen des Produkts die Schritte umfasst:

a) Einrbringen des Produkts in einen Trockenofen, dessen Temperatur auf mindestens 90°C, vorzugsweise mindestens 100°C, erhöht wird, und der Ofen bei dieser Temperatur gehalten wird, bis das Celluloseprodukt mindestens ungefähr die gleiche Temperatur erreicht hat,
b) anschließend allmähliches Erhöhen der Temperatur des Ofens, wobei der Unterschied zwischen der Innentemperatur der Produkte und der Ofentemperatur bei weniger als 30°C gehalten wird, bis der gewünschte Feuchtigkeitsgehalt des Holzes erreicht worden ist, und schließlich
c) gegebenenfalls allmähliches Senken der Ofentemperatur, wobei der Unterschied zwischen der Innentemperatur des Holzes und der Ofentemperatur bei weniger als 30°C gehalten wird, bis die Innentemperatur des Holzes den gewünschten Wert erreicht hat.

5. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass das auf einen Feuchtigkeitsgehalt von weniger als 15% getrocknete Celluloseprodukt 1 bis 20 Std., vorzugsweise 2 bis 10 Std., bei 180 bis 250°C behandelt wird.

6. Verfahren nach einem der vorstehenden Ansprüche, dadurch gekennzeichnet, dass dem Verfahren gesättigter Wasserdampf zugeführt wird.

7. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass Holz, Pfosten, Holzstämme, Schnittholz, Furnier, Sperrholz, Späne, Sägemehl oder Fasern behandelt werden.

8. Verfahren nach Anspruch 1, wobei Kiefernschnittholz behandelt wird, dadurch gekennzeichnet, dass das getrocknete Schnittholz 2 bis 8 Std. bei einer Temperatur von 200 bis 250°C gehalten wird, so dass ein guter Schutz gegen Fäulnis erzielt wird.

9. Verfahren nach Anspruch 1, wobei Fichtenschnittholz behandelt wird, dadurch gekennzeichnet, dass das getrocknete Schnittholz 2 bis 8 Std. bei einer Temperatur von 175 bis 210°C gehalten wird, so dass ein guter Schutz gegen Fäulnis erzielt wird.

10. Verfahren nach Anspruch 1, wobei Birkenholz behandelt wird, dadurch gekennzeichnet, dass das getrocknete Schnittholz 2 bis 8 Std. bei einer Temperatur von 200 bis 250°C gehalten wird, so dass ein guter Schutz gegen

Fäulnis erzielt wird.

11. Verfahren nach Anspruch 1, wobei Lärchenholz behandelt wird, dadurch gekennzeichnet, dass das getrocknete Schnittholz 2 bis 8 Std. bei einer Temperatur von 200 bis 250°C gehalten wird, so dass ein guter Schutz gegen Fäulnis erzielt wird.

Revendications

1. Procédé d'augmentation de la résistance de produits dérivés de cellulose au pourrissement et à la désagrégation et d'amélioration de la stabilité dimensionnelle du produit, procédé selon lequel :

   - les produits dérivés de cellulose sont séchés jusqu'à une teneur en humidité de moins de 15 %,
   - ils sont soumis à un traitement thermique exécuté à une température augmentée,

   caractérisé en ce que

   - après séchage, les produits sont conservés à sensiblement la pression atmosphérique à une température au-dessus de 150 °C dans un four humide, auquel de la vapeur d'eau est amenée, et
   - le traitement est continué jusqu'à ce qu'une perte de poids d'au moins 5 % ait lieu dans le produit.

2. Procédé selon la revendication 1, dans lequel les produits dérivés de cellulose sont séchés à une température élevée, caractérisé en ce que, pendant le séchage du produit mouillé, la différence entre les températures intérieure et extérieure du produit est maintenue à environ 10 à 30 °C afin d'empêcher la fissuration.

3. Procédé selon la revendication 2, caractérisé en ce que le produit est séché en présence de vapeur.

4. Procédé selon la revendication 2 ou 3, caractérisé en ce que le séchage du produit comprend les étapes suivantes :

   a) son placement dans un four de séchage dont la température est élevée à au moins 90 °C, de préférence à au moins 100 °C, et le four est conservé à cette température jusqu'à ce que le produit dérivé de cellulose ait au moins à peu près atteint la même température,
   b) ensuite, augmentation progressive de la température du four tout en maintenant la différence entre la température intérieure des produits et la température du four à moins de 30 °C jusqu'à ce que la teneur en humidité souhaitée du bois ait été atteinte, et finalement
   c) abaissement progressif facultatif de la température du four tout en conservant la différence entre la température intérieure du bois et la température du four à moins de 30 °C jusqu'à ce que la température du bois ait atteint la valeur souhaitée.

5. Procédé selon l'une quelconque des revendications précédentes, caractérisé en ce qu'un produit dérivé de cellulose séché jusqu'à une teneur en humidité de moins de 15 % est traité à 180 à 250 °C pendant 1 à 20 heures, de préférence pendant environ 2 à 10 heures.

6. Procédé selon l'une quelconque des revendications précédentes, caractérisé en ce que de la vapeur d'eau saturée est amenée au processus.

7. Procédé selon la revendication 1, caractérisé par le traitement de bois, de poteaux, de montants, de bois scié, de placage, de contreplaqué, de copeaux, de sciure, ou de fibres.

8. Procédé selon la revendication 1, dans lequel du bois de pin scié est traité, caractérisé par la conservation du bois scié séché à une température de 200 à 250 °C pendant 2 à 8 heures de façon à parvenir à une bonne protection contre le pourrissement.

9. Procédé selon la revendication 1, dans lequel du bois de sapin scié est traité, caractérisé par la conservation du bois scié séché à une température de 175 à 210 °C pendant 2 à 8 heures de façon à parvenir à une bonne protection contre le pourrissement.

10. Procédé selon la revendication 1, dans lequel une matière de bois de bouleau est traitée, caractérisé par la con-

servation du bois scié séché à une température de 200 à 250 °C pendant 2 à 8 heures de façon à parvenir à une bonne protection contre le pourrissement.

11. Procédé selon la revendication 1, dans lequel une matière de bois de mélèze est traitée, caractérisé par la conservation du bois scié séché à une température de 200 à 250 °C pendant 2 à 8 heures de façon à parvenir à une bonne protection contre le pourrissement.

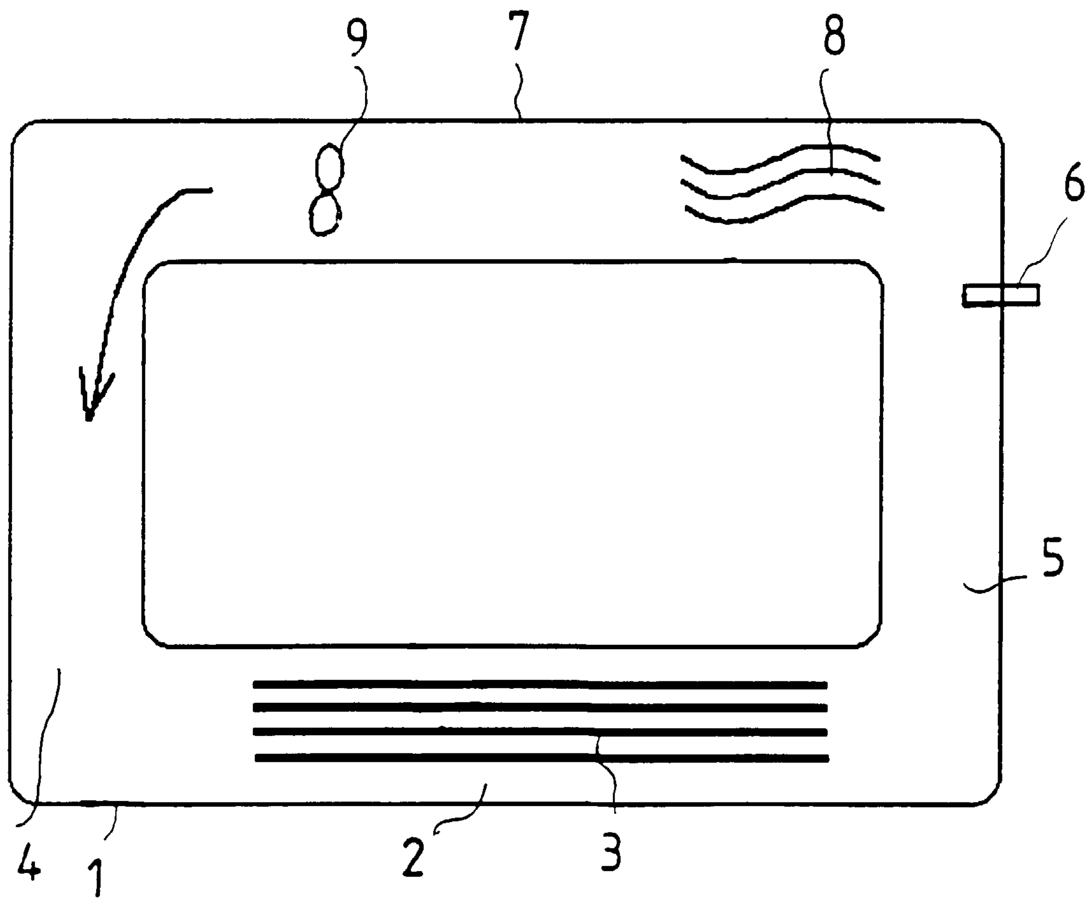

Fig. 1

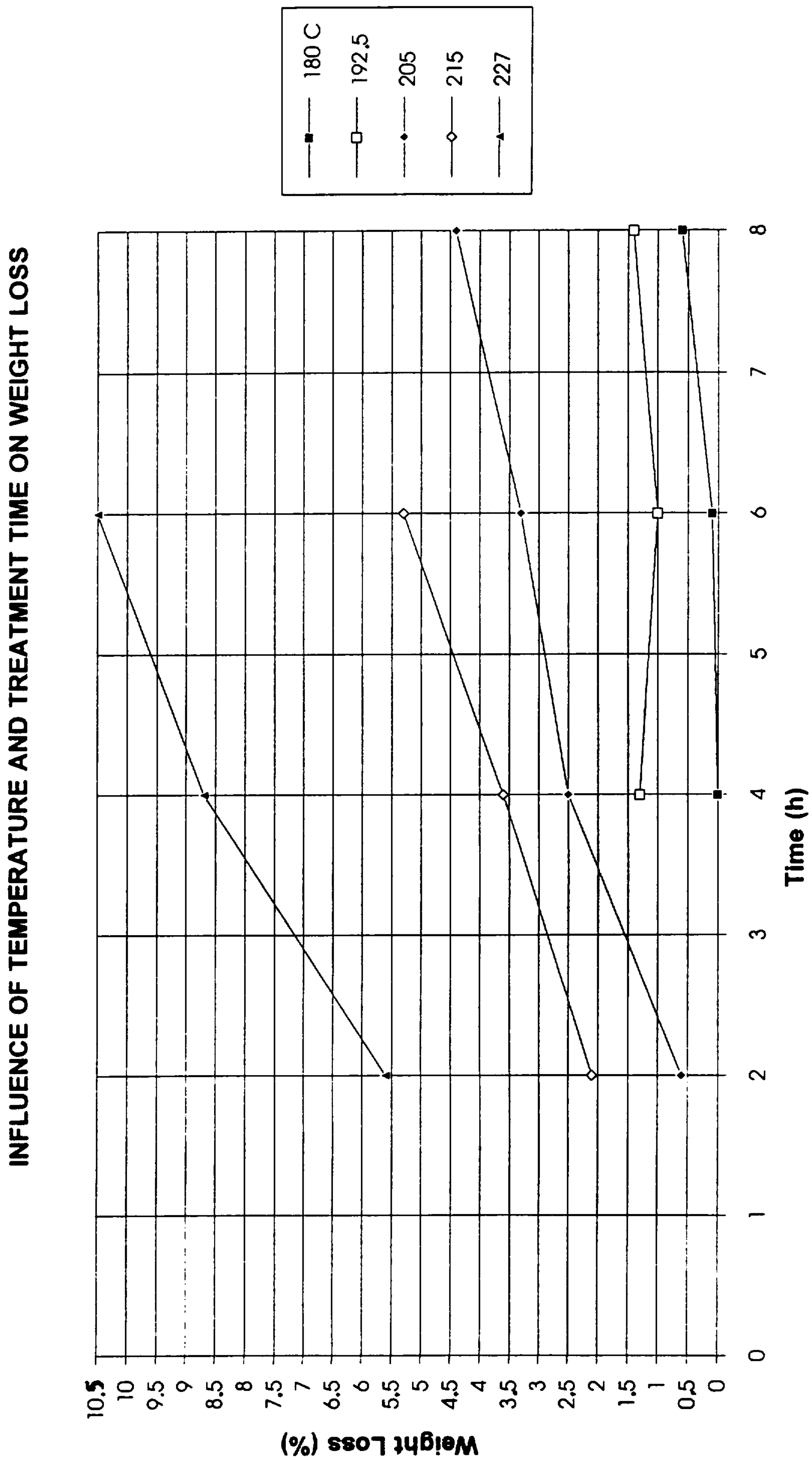
INFLUENCE OF TEMPERATURE AND TREATMENT TIME ON WEIGHT LOSS
180 C
192,5
205
215
227
Weight Loss (%)
10,5
10
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
0
Time (h)
0
1
2
3
4
5
6
7
8

Fig. 2

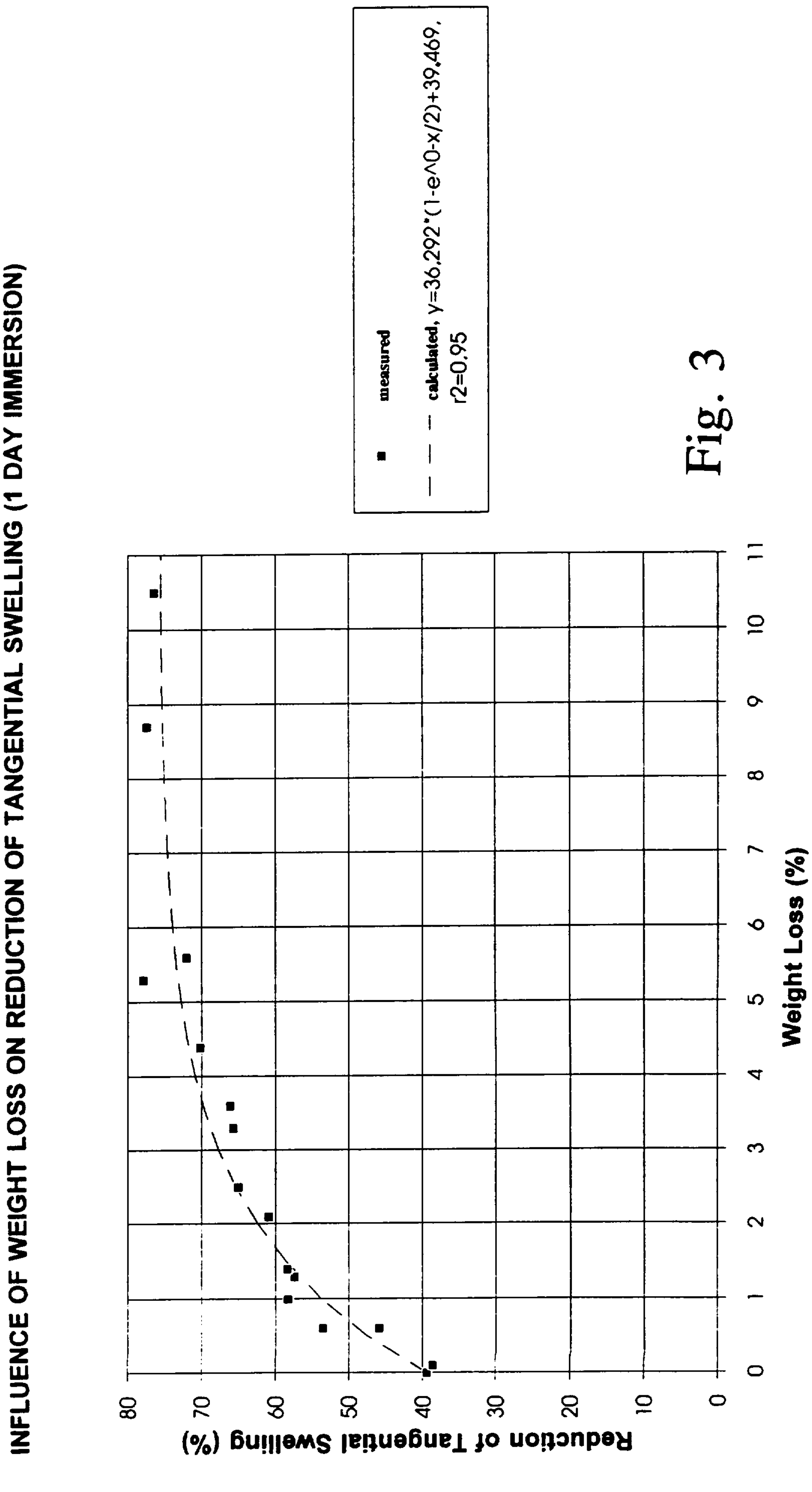
INFLUENCE OF WEIGHT LOSS ON REDUCTION OF TANGENTIAL SWELLING (1 DAY IMMERSION)
Reduction of Tangential Swelling (%)
0
10
20
30
40
50
60
70
80
Weight Loss (%)
0
1
2
3
4
5
6
7
8
9
10
11
measured
calculated, y=36,292*(1-e^0-x/2)+39,469, r2=0,95

Fig. 3

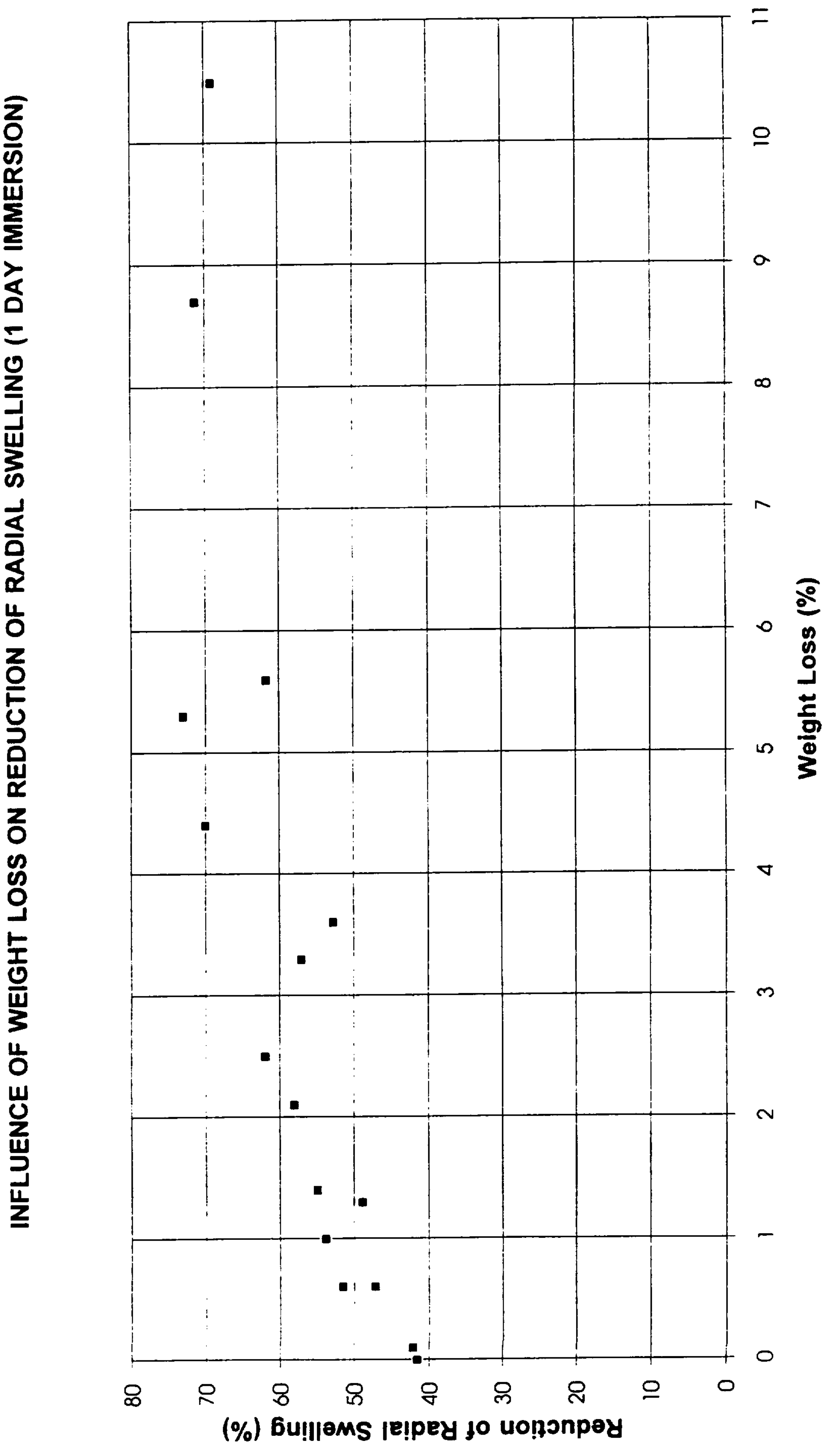
INFLUENCE OF WEIGHT LOSS ON REDUCTION OF RADIAL SWELLING (1 DAY IMMERSION)
Reduction of Radial Swelling (%)
0
10
20
30
40
50
60
70
80
Weight Loss (%)
0
1
2
3
4
5
6
7
8
9
10
11

Fig. 4

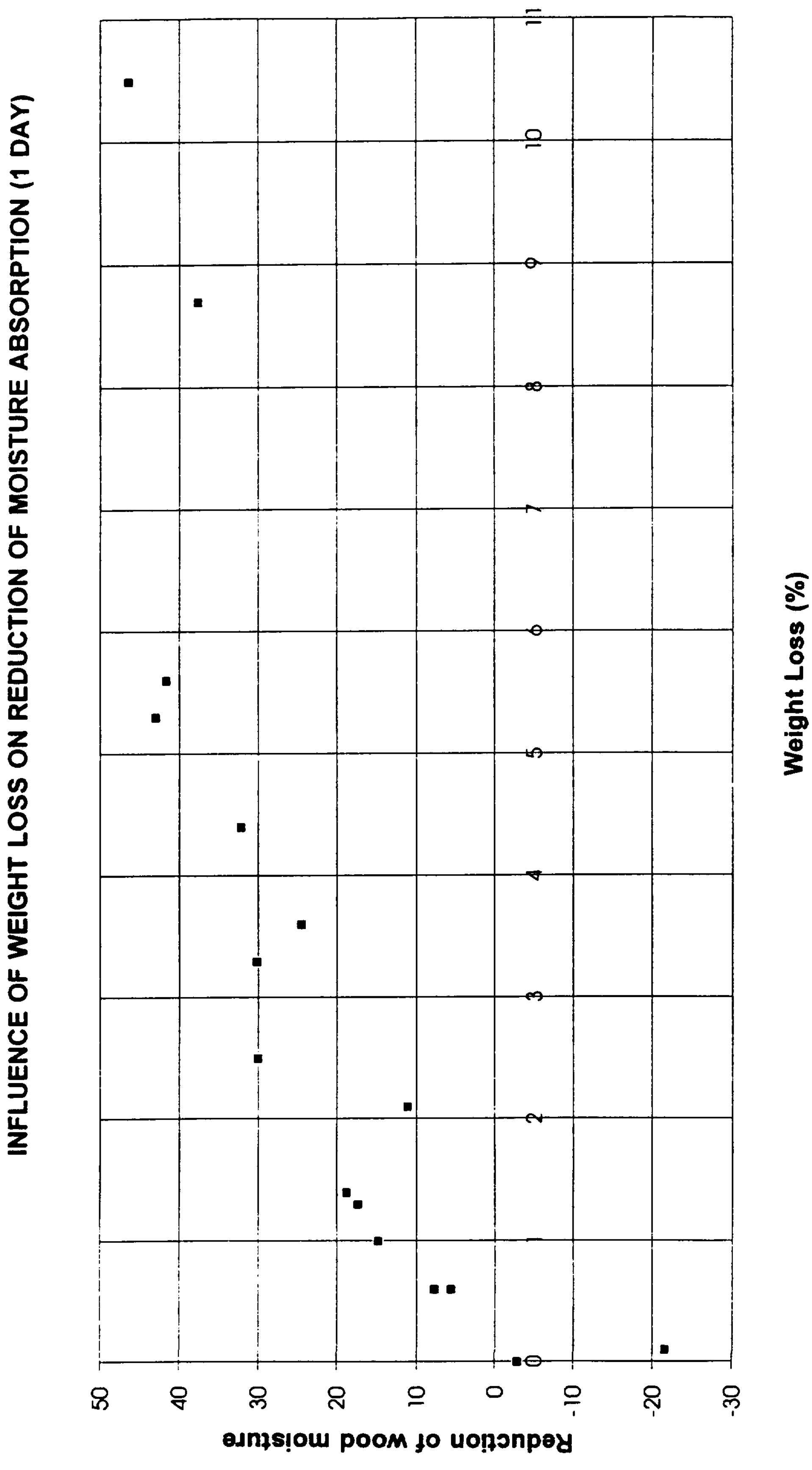
INFLUENCE OF WEIGHT LOSS ON REDUCTION OF MOISTURE ABSORPTION (1 DAY)
Reduction of wood moisture
50
40
30
20
10
0
-10
-20
-30
Weight Loss (%)
0
1
2
3
4
5
6
7
8
9
10
11

Fig. 5

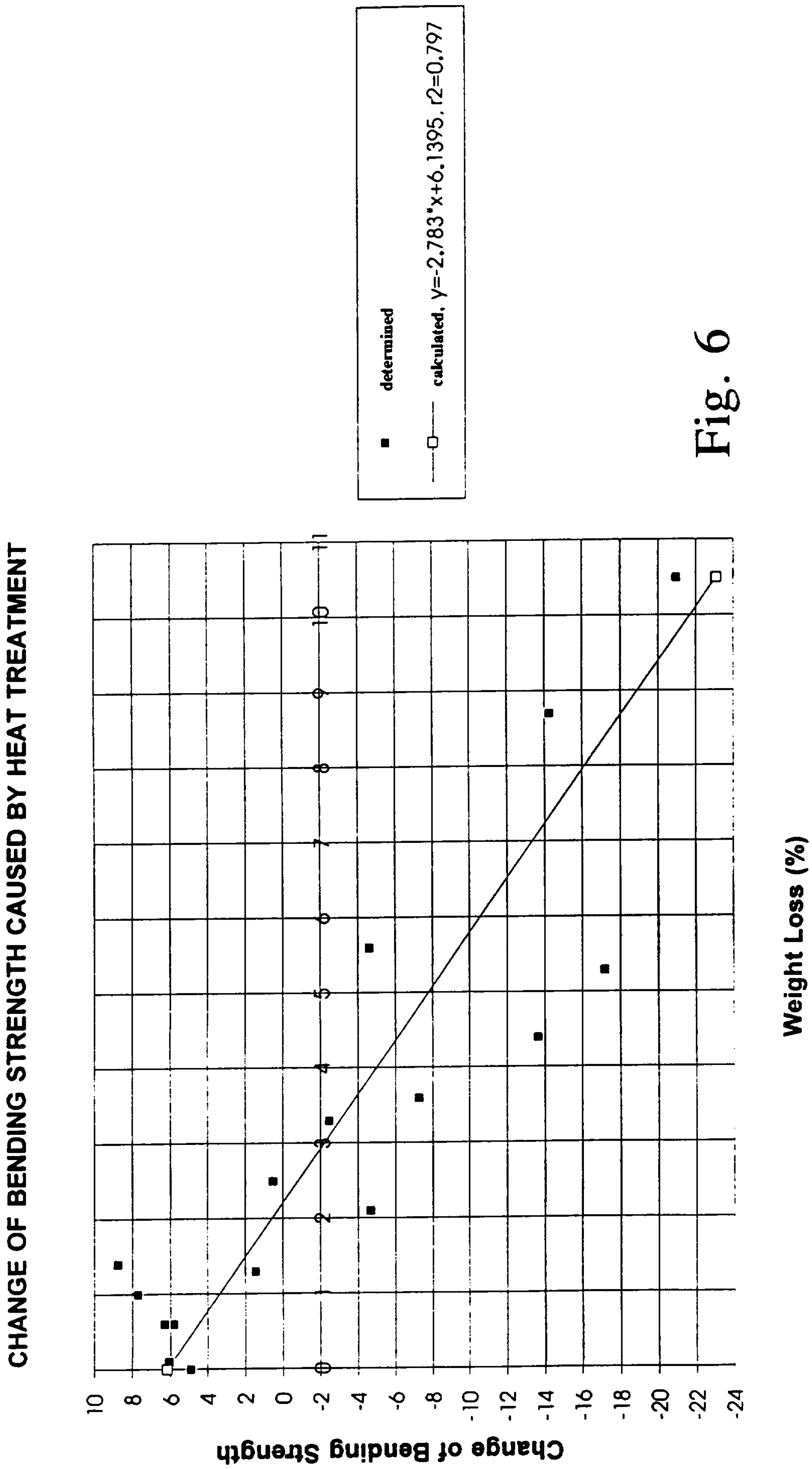
CHANGE OF BENDING STRENGTH CAUSED BY HEAT TREATMENT
Change of Bending Strength
Weight Loss (%)
10
8
6
4
2
0
-2
-4
-6
-8
-10
-12
-14
-16
-18
-20
-22
-24
0
1
2
3
4
5
6
7
8
9
10
11
determined
calculated, y=-2,783*x+6,1395, r2=0,797

Fig. 6

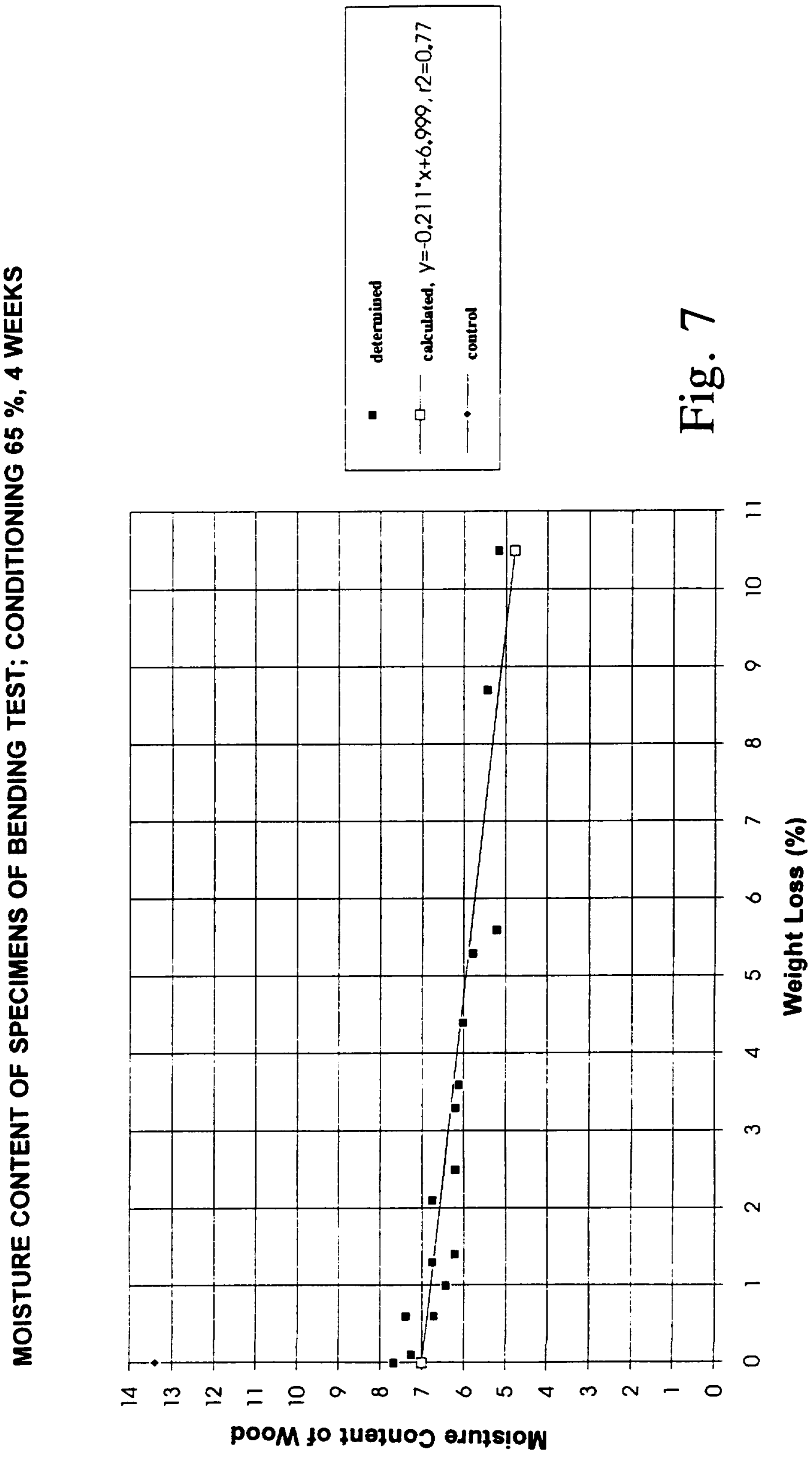
MOISTURE CONTENT OF SPECIMENS OF BENDING TEST; CONDITIONING 65 %, 4 WEEKS
Moisture Content of Wood
Weight Loss (%)
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14
0 1 2 3 4 5 6 7 8 9 10 11
determined
calculated, y=-0,211*x+6,999, r2=0,77
control

Fig. 7

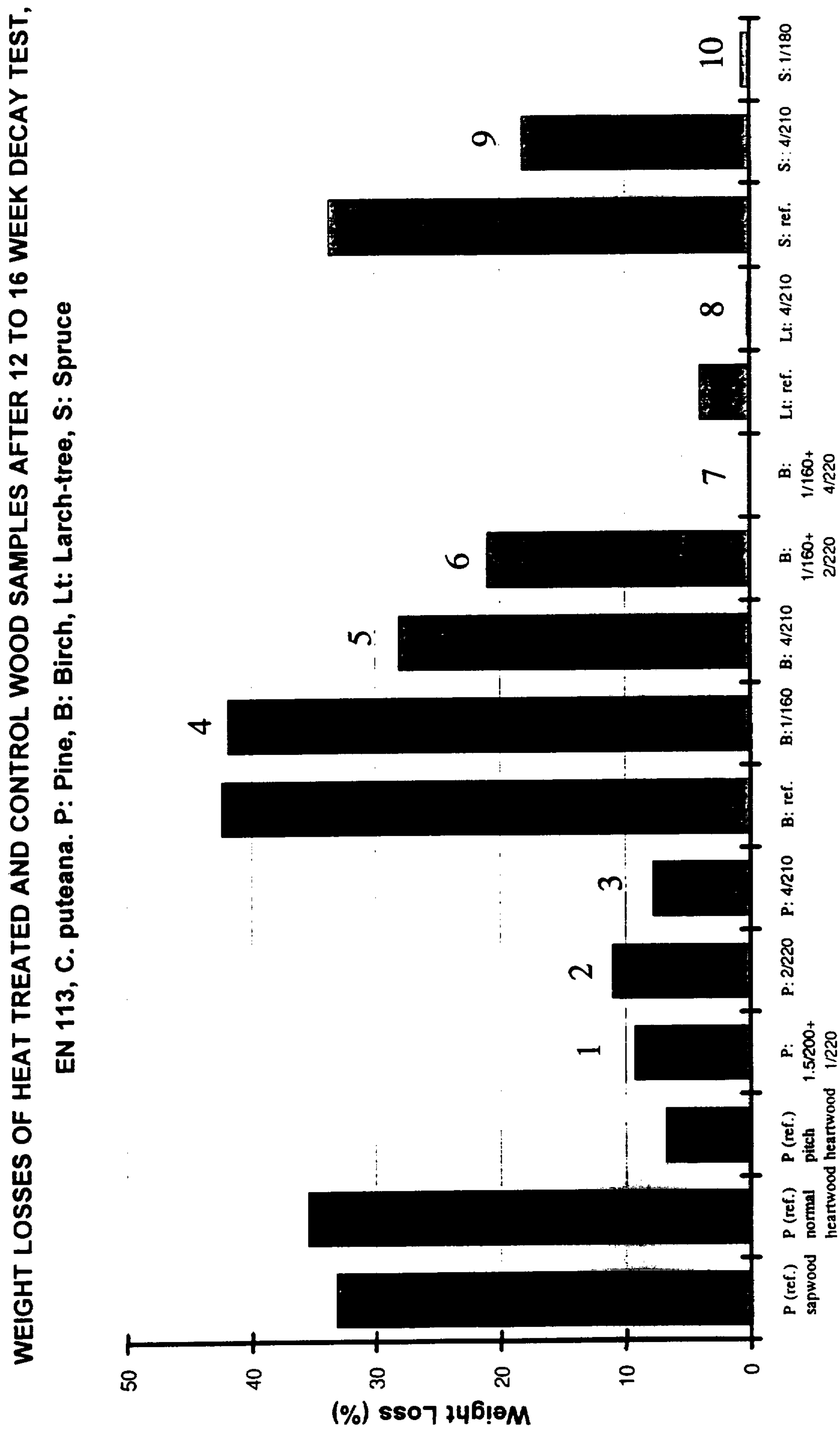
WEIGHT LOSSES OF HEAT TREATED AND CONTROL WOOD SAMPLES AFTER 12 TO 16 WEEK DECAY TEST,
EN 113, C. puteana. P: Pine, B: Birch, Lt: Larch-tree, S: Spruce
Weight Loss (%)
0
10
20
30
40
50
P (ref.) sapwood
P (ref.) normal heartwood
P (ref.) pitch heartwood
P: 1.5/200+ 1/220
P: 2/220
P: 4/210
B: ref.
B: 1/160
B: 4/210
B: 1/160+ 2/220
B: 1/160+ 4/220
Lt: ref.
Lt: 4/210
S: ref.
S: 4/210
S: 1/180
1
2
3
4
5
6
7
8
9
10

Fig. 8

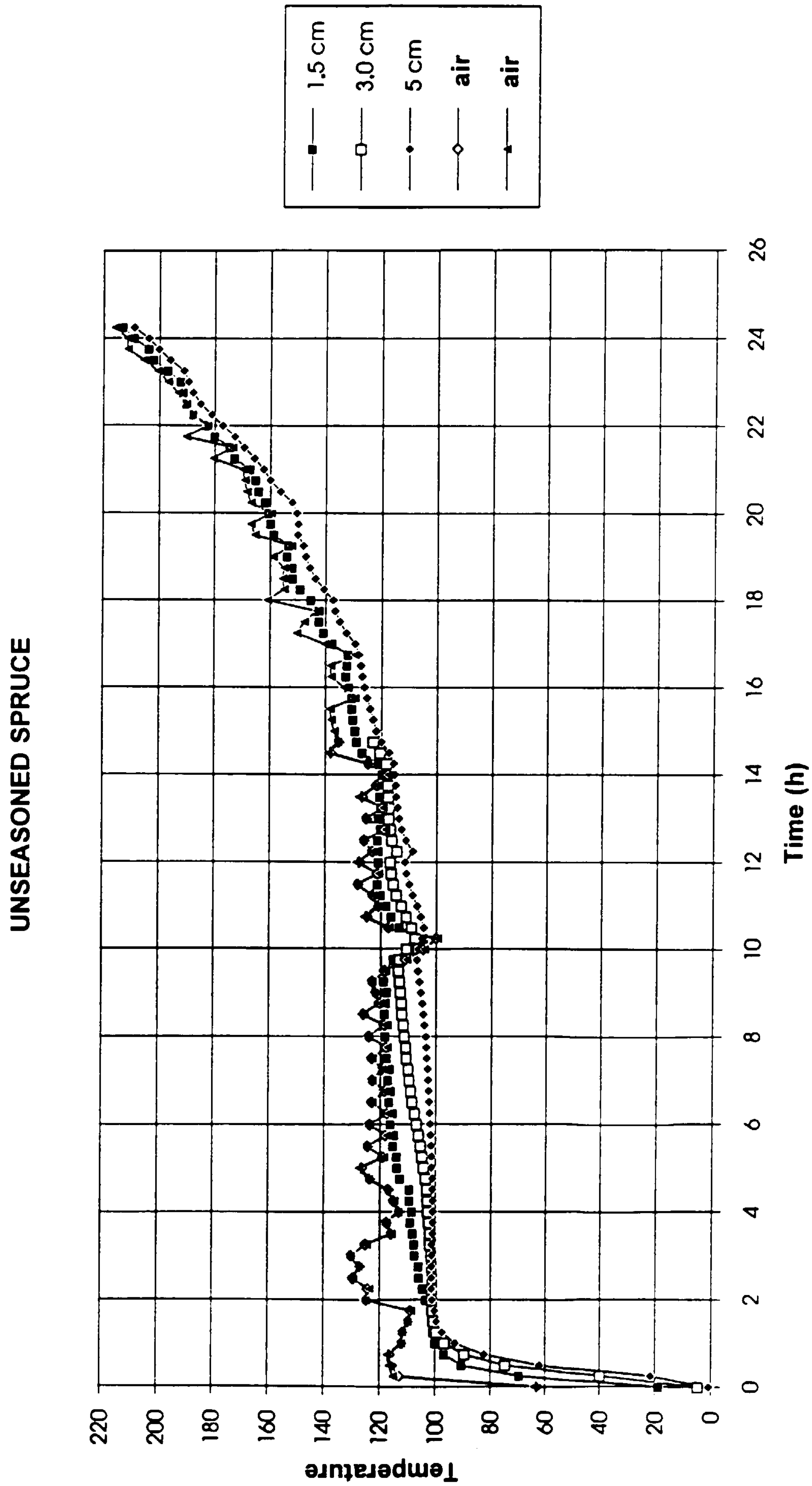
UNSEASONED SPRUCE
Temperature
0
20
40
60
80
100
120
140
160
180
200
220
Time (h)
0
2
4
6
8
10
12
14
16
18
20
22
24
26
1.5 cm
3.0 cm
5 cm
air
air

Fig. 9

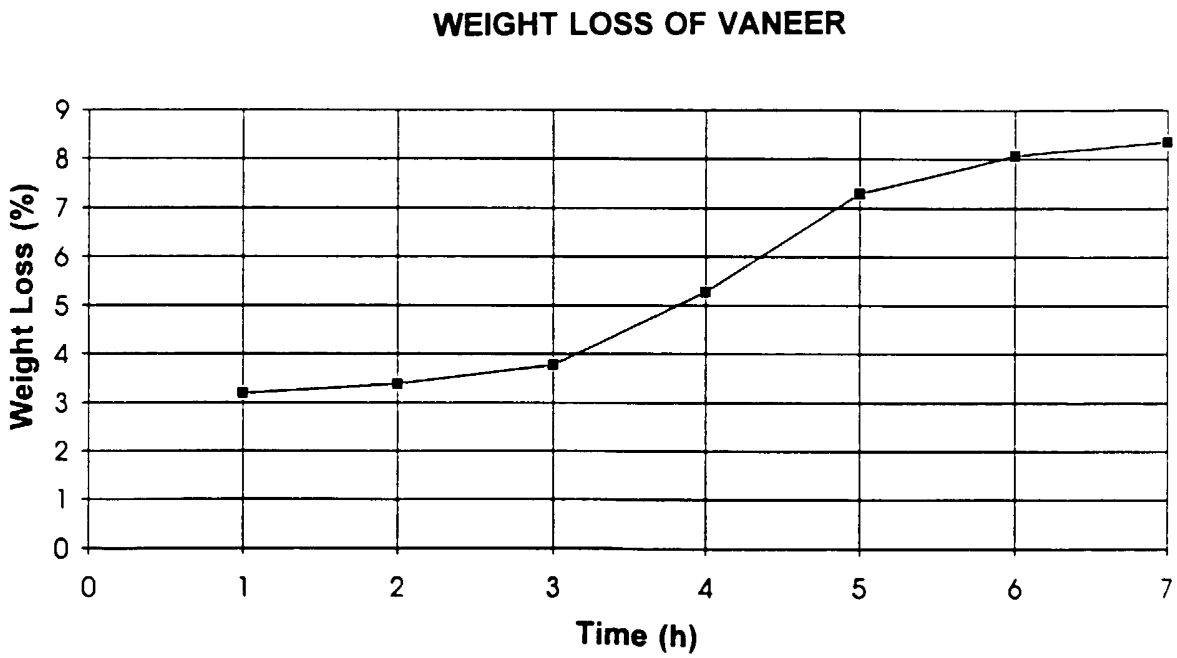
WEIGHT LOSS OF VANEER
Weight Loss (%)
9
8
7
6
5
4
3
2
1
0
0
1
2
3
4
5
6
7
Time (h)

Fig. 10

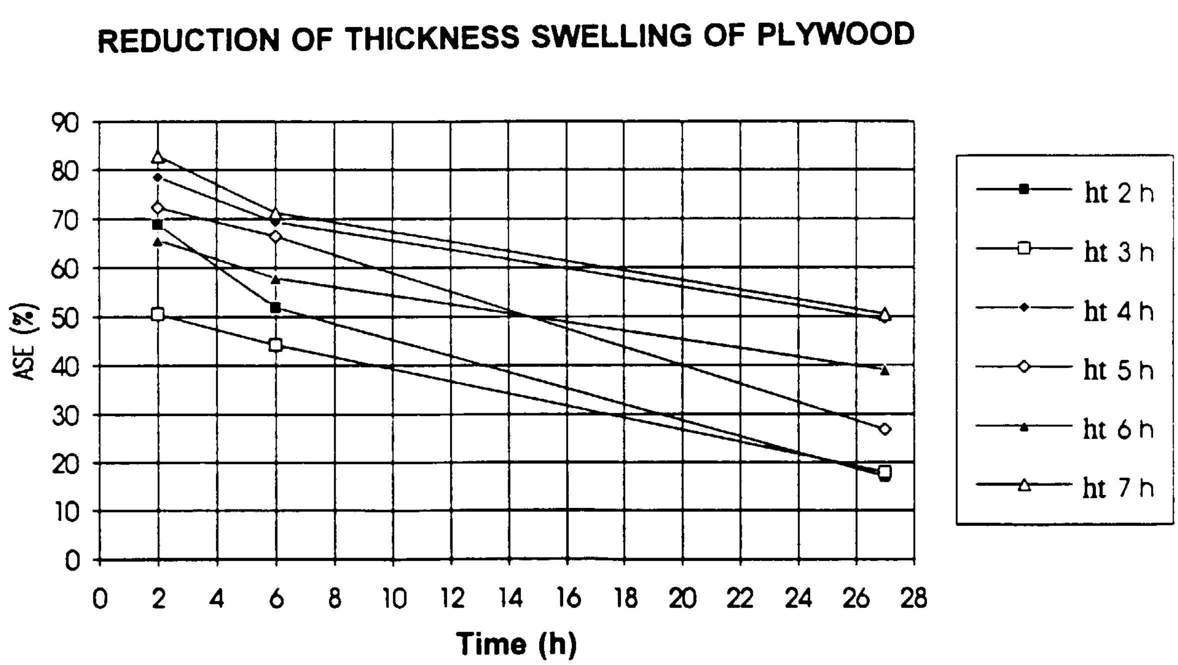
REDUCTION OF THICKNESS SWELLING OF PLYWOOD
ASE (%)
90
80
70
60
50
40
30
20
10
0
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
Time (h)
ht 2 h
ht 3 h
ht 4 h
ht 5 h
ht 6 h
ht 7 h

Fig. 11

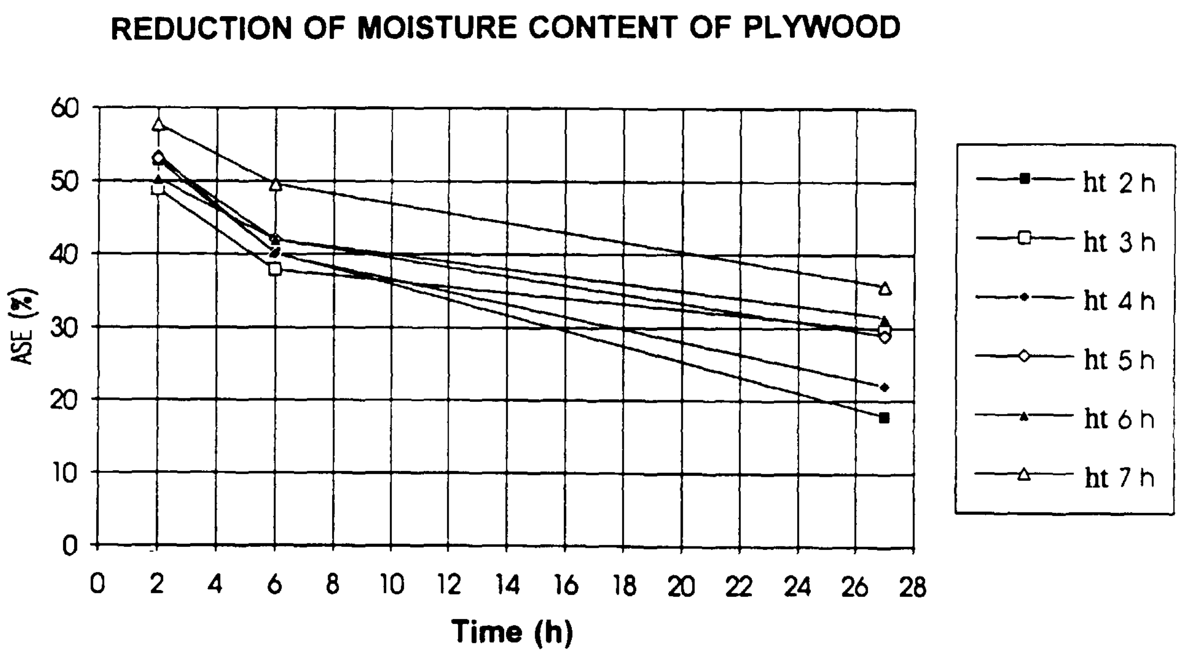
REDUCTION OF MOISTURE CONTENT OF PLYWOOD
ASE (%)
0
10
20
30
40
50
60
0 2 4 6 8 10 12 14 16 18 20 22 24 26 28
Time (h)
ht 2 h
ht 3 h
ht 4 h
ht 5 h
ht 6 h
ht 7 h

Fig. 12